Relative dating of the fossil hominids of Europe

K. P. Oakley, F.B.A.

2 Islip Place, Oxford OX2 7SR

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Synopsis

The application of analysis of skeletal materials for fluorine, 'uranium' and nitrogen contents is described as a method of relative dating, with particular reference to Pleistocene and early Post-Pleistocene hominids and associated Mammalia in Europe. Tables of analyses are presented.

Introduction

When there is doubt as to whether a bone[1] or tooth is contemporaneous with other skeletal remains in a particular deposit, techniques are now available which in favourable circumstances can solve the problem. The fluorine and 'uranium' contents of the mineral matter of buried bones and teeth increase with the passage of time, whereas the organic (protein) content measured as nitrogen decreases. Thus, comparison of the fluorine, 'uranium' and nitrogen contents of a bone or tooth of questionable age with the ranges of these elements present in other bones or teeth of known age and in similar matrix at the same site may indicate clearly the relative antiquity of the specimen in question.

Whether the comparison of fluorine content, of 'uranium' content or of nitrogen content is the more likely to solve a particular bone or tooth dating problem, or whether the use of two of these methods or of all three in combination is necessary or preferable, will depend on the local circumstances. These methods are essentially empirical. As they are methods of relative and not

[1] In reading this Introduction it should be understood that the relative dating methods applicable to 'bone' are equally applicable to 'antler'. Strictly speaking therefore any reference to bone should be read as 'bone or antler'.

absolute dating, it is essential to have local controls. It would be impossible to date a bone or tooth relatively by fluorine, 'uranium' or nitrogen analysis if it had been found at a site which had yielded no skeletal material of known age for analytical comparison, although if both the 'uranium' and the fluorine content of an isolated bone or tooth were high and the nitrogen low one might infer that the specimen was 'fossil' rather than Recent.

History of the fluorine dating method

At the beginning of the nineteenth century the Italian chemist Morichini (1805) detected fluorine (in the form of 'fluate of lime') in the enamel of a fossil elephant tooth found near Rome. Gay-Lussac (1806), commenting on this result, said that it was a discovery which might have important consequences. However, he thought that the 'fluoric acid' had been absorbed by the animal during its life. Two other French chemists, Fourcroy & Vauquelin (1806a, b), reported that they had failed to find fluorine in a new ivory or enamel, whereas they confirmed its presence in fossil ivories. 'This singular circumstance' they said 'seems to indicate that fluoric acid exists in the earth; . . . that during the long continuance of these substances [ivories] in the earth they combine with the fluoric acid'.

With refinements in methods of analysis it became clear that new ivory or dentine and bone contain minute traces of fluorine but that after fossilization they contain considerably more, having adsorbed it from percolating water or the soil in which they have been embedded. An English chemist, James Middleton (1844), was the first to recognize this fact clearly, and he read a paper to the Geological Society of London, showing that fossil bones contained fluoride in proportion to their antiquity. He compared the fluorine content of various fossil bones with that of a Greek human skeleton 2 000 years old. He estimated on this basis that fossil mammal bones from the Siwalik beds of India had an antiquity of 7 700 years, and one from the Eocene beds in France, of 24 000 years. Middleton's brave attempt at geochronology was not taken seriously, and his discovery appears to have been forgotten for a century or so, but the principle was reaffirmed fifty years later by a French mineralogist, Adolphe Carnot (1892a, 1893). He analysed a number of fossil bones obtained from different localities, representing geological horizons ranging from Ordovician to Recent. By averaging the results he showed conclusively that the concentration of fluorine in fossil bones varies almost uniformly with time. Carnot expressed the fluorine content of the analysed bones as the proportion relative to that in fluorapatite taken as unity. If we assume the maximum fluorine content of fluorapatite is 3.8%, his figures can be converted into average percentages of fluorine in bones of successive geological epochs as follows:

Recent	< 0.3
Pleistocene	1.5
Tertiary	2.3
Mesozoic	3.4
Palaeozoic	3.7

By considering Carnot's individual analyses it is evident that the averaging process has obscured the important fact that there is a wide variation in the concentration of fluorine in bones of the same geological age but from different localities. So many variables are involved that it would be impossible to ascertain accurately the geological age of any isolated fossil by determining how much fluorine it contained; still less would it be possible to calculate the absolute age of a vertebrate specimen from its fluorine content. At one locality fluorine may be abundant in the ground-water, at another it may be only present as a trace. The rate at which fluorine is accumulated in skeletal material also depends on climatic factors and on the permeability of the matrix. For all these reasons, Carnot's work was generally regarded as interesting but as having no practical outcome, and like Middleton's paper entered the scientific limbo.

It is difficult to account for the importance of Carnot's discoveries being overlooked, particularly as another paper by him (Carnot 1892b) contained the essence of the method of relative dating by fluorine which was revived with considerable success in the present century. He showed

that by analysing the fluorine content of a human bone found in a Pleistocene gravel pit at Billancourt (Seine) and comparing it with the fluorine content of fossil bones from the same site, the human bone evidently represented an intrusive burial of Post-Pleistocene age. The following figures illustrate the striking difference between the degree of fluorination in the Pleistocene and in the Post-Pleistocene material at Billancourt:

Human tibia	Pleistocene mammal bones
0·17% F	1·43% F
	1·84% F

Only a few of Carnot's contemporaries realized the potentialities of his results, but one was Thomas Wilson, Curator of Prehistoric Archeology in the U.S. National Museum, who had the 'fluorine test' applied to the pelvic bone of the Natchez human skeleton in Mississippi, thereby proving that it has the same antiquity as the associated bone of the Pleistocene ground-sloth *Mylodon* (Wilson 1902, Stewart 1951).

During World War II, while I was working with the Geological Survey of Great Britain, research into phosphate resources and the geological aspects of dental fluorosis (Bromehead 1943; Oakley 1943) focussed our attention on Carnot's forgotten papers. It became evident that if one were interested in separating bones of different ages which happened to have been mixed together in a single deposit, either through artificial interment or through natural rearrangement such as by stream action, widely different concentrations of fluorine in the bones could be most revealing. This is the theoretical basis of the fluorine-dating method.

In 1947 Dr H. J. Walls, then in the Home Office Forensic Science Laboratory at Bristol, undertook to determine the fluorine content of a series of selected bone and tooth samples which I obtained, with the cooperation of the late Dr L. S. B. Leakey, from various deposits in the region of the Kavirondo Gulf on the Kenya side of Lake Victoria. Working with samples weighing 20–40 mg, Walls used the method of determining fluorine described by Milton, Lidell & Chivers (1947). It was hoped that the results might settle the questions regarding the antiquity of the fossil human bones from Kanam and Kanjera (Boswell 1935). In fact they showed clearly that the method was not applicable in regions where fluorine is excessively abundant in the ground-water, as it is in most volcanic areas with tropical weathering, where fluorination of vertebrate materials occurs rapidly and sometimes in a random fashion. The analyses of the East African material reported by Walls may be summarized as follows:

Fossil bones from Kavirondo Gulf	Fluorine content
Upper Pleistocene and Holocene bones (5)	1·2–4·9%
Middle Pleistocene bones (6)	0·5–3·4%
Lower Pleistocene bones (2)	1·7–3·4%

These results appeared to contradict Carnot's rule, but if a larger number of fossil bones and teeth from the same sites had been analysed this might not have been the case. However, the fact remains that in pyroclastic sediments, such as those abounding in East Africa, volcanic ashes rich in fluorine sometimes contribute to the formation of layers overlying deposits in which the fluorine content is much lower. This would account for the fact that the average fluorine content of bone samples from Bed IV in Olduvai Gorge, Tanzania, is higher than that of bone samples from the underlying Bed III (Day & Molleson 1976 : 456).

It is remarkable that some Upper Pleistocene bones in the Kavirondo Gulf series (notably from Kuguta) contained over 5% fluorine, greatly in excess of the theoretical maximum for fluorapatite (3·8%). Under exceptional geochemical conditions the isomorphously related sodium fluorapatite $(Ca_6Na_4(PO_3F)_6O_2;$ Mehta & Simpson 1975) may be formed and in this the fluorine content could theoretically be as high as 12% (*fide* Duncan McConnell, Emeritus Professor, The Ohio State University, *in lit.* Oct. 27, 1976). However, it should be noted that according to Glover & Phillips (1965 : 574) in some of the oldest bone samples analysed in the BM(NH) programme, where a

greater proportion of fluorine was found than that required in fluorapatite, X-ray diffraction evidence suggested that some fluorine may be present as calcium fluoride in the bone matrix. Mineralogical investigation of the fluorine-rich bones collected by Leakey from Kuguta near Homa Mountain, Kenya is clearly desirable.

The fluorine content of the controversial Kanam mandible ranged from 1·4% to 2·2%, but strangely enough its fluorine/phosphate ratio (as defined on p. 5) was at one point 30, which is higher than that recorded in any other fossil bone analysed in our programme of work. Although this jaw was presumed when discovered (Leakey 1932 : 722) to have been contemporaneous with the Lower Pleistocene deposit in which it appeared to lie, comparative analyses eventually indicated that it was probably in an intrusive calcrete block, and no older than Middle Pleistocene (Oakley 1975).

There seemed a much better chance that the method of fluorine dating would yield more useful results in regions of temperate weathering, where one might reasonably expect the increase of fluorine in fossil bones and teeth to be gradual. In 1948 the then Department of the Government Chemist in London agreed to cooperate with the British Museum (Natural History) in exploring more fully the possible applications of this method. The initial aim was to confirm or disprove the alleged antiquity of the Galley Hill skeleton (for results of this enquiry see p. 17). At the same time the antiquity of bones of the Swanscombe skull was confirmed by this method (see p. 26).

With the aid of grants from the Wenner-Gren Foundation, the Subdepartment of Anthropology of the BM(NH) has had about a thousand vertebrate specimens selectively analysed. The results have been used in preparing this Introduction. Early in the investigations the analyses for fluorine content of the vertebrate samples were made in the Department of the Government Chemist by R. H. Settle, E. C. W. Maycock and C. R. Hoskins, using a method similar to that described by Willard & Winter (1933). Later when the fluorine dating method was applied to the Piltdown bones and teeth the analytical work was carried out by Hoskins & Fryd (1955). They worked with 20–100 mg samples when available, but in exceptional cases they made determinations of fluorine in samples weighing considerably less than 20 mg. Continuation of analytical work on fossil bones and teeth was carried out by Glover & Phillips (1965).

At this stage it seems appropriate to sum up the principles of the fluorine dating method. As Middleton found, buried bones and teeth adsorb fluorine from ground-water or from moist sedimentary matrices, and by a process of irreversible ionic interchange the element is fixed in their mineral substance. Consequently when bones or teeth of different ages occur at the same site, comparison of their fluorine contents provides a useful method of estimating their relative antiquity. The main mineral constituent of bones and teeth, hydroxyapatite $Ca_{10}(PO_4)_6.(OH)_2$, has a strong affinity for fluorine which in the form of fluorides occurs as a trace in the ground-water of sedimentary formation and soils, usually in the proportion of about one part in a million. When fluoride ions come into contact with this mineral matter they are adsorbed and locked into its structure, in fact displacing the hydroxyl ions in the crystal lattice of the hydroxyapatite, which is thus converted particle by particle into fluorapatite $Ca_{10}(PO_4)_6.F_2$. This form of apatite is more stable and is not readily dissolved, so that unless conditions in the soil or rock formation become so acid that the whole vertebrate specimen is destroyed the fluoride ions which have entered its structure are not removed. Owing to both the ease with which fluoride ions diffuse and the porosity of bony material, the fixation of the fluorine is not confined to the surface of the specimen but takes place relatively uniformly throughout its substance, at any rate under temperate conditions of weathering; see analyses of the fossil deer antler from Swanscombe quoted below. All types of bone, antler, and dentine are nearly equal in their capacity for adsorbing fluorine, but the enamel of teeth is more resistant to the penetration of fluoride ions, especially in the early stage of fossilization. The theoretical maximum fluorine content of fossil bones or teeth is 3·8%.

In uniformly porous dentine, bone and antler there is very little evidence of zonation of the adsorbed fluorine. Apparent differences in fluorine content between the outside and the inside of a bone, tooth or antler occur sometimes because the interior tissue is more cancellar (spongy) and consequently often contains grains of infiltrated mineral matter. Thus, the degree of fluorination of a bone or other vertebrate specimen is more usefully expressed by the fluorine/phosphate ratio

than by the percentage of fluorine, as the following example illustrates; cross-section of a Pleistocene cervid antler, BM(NH) E.600:

	F%	P_2O_5%	$\dfrac{F\%}{P_2O_5\%} \times 100$
Outer zone, compact tissue	1·2	26·0	4·6
Inner zone, spongy tissue (with infiltrated clay)	0·2	5·0	4·0

If comparison is made between the fluorine/phosphate ratio rather than between the fluorine content of the samples the complicating factor of any contamination by adventitious mineral matter is eliminated. Although the ratio is usually written out as

$$\frac{F\%}{P_2O_5\%} \times 100$$

for convenience it can be stated as $100F/P_2O_5$. This practice was followed by Oakley, Campbell & Molleson (1971 : ix).
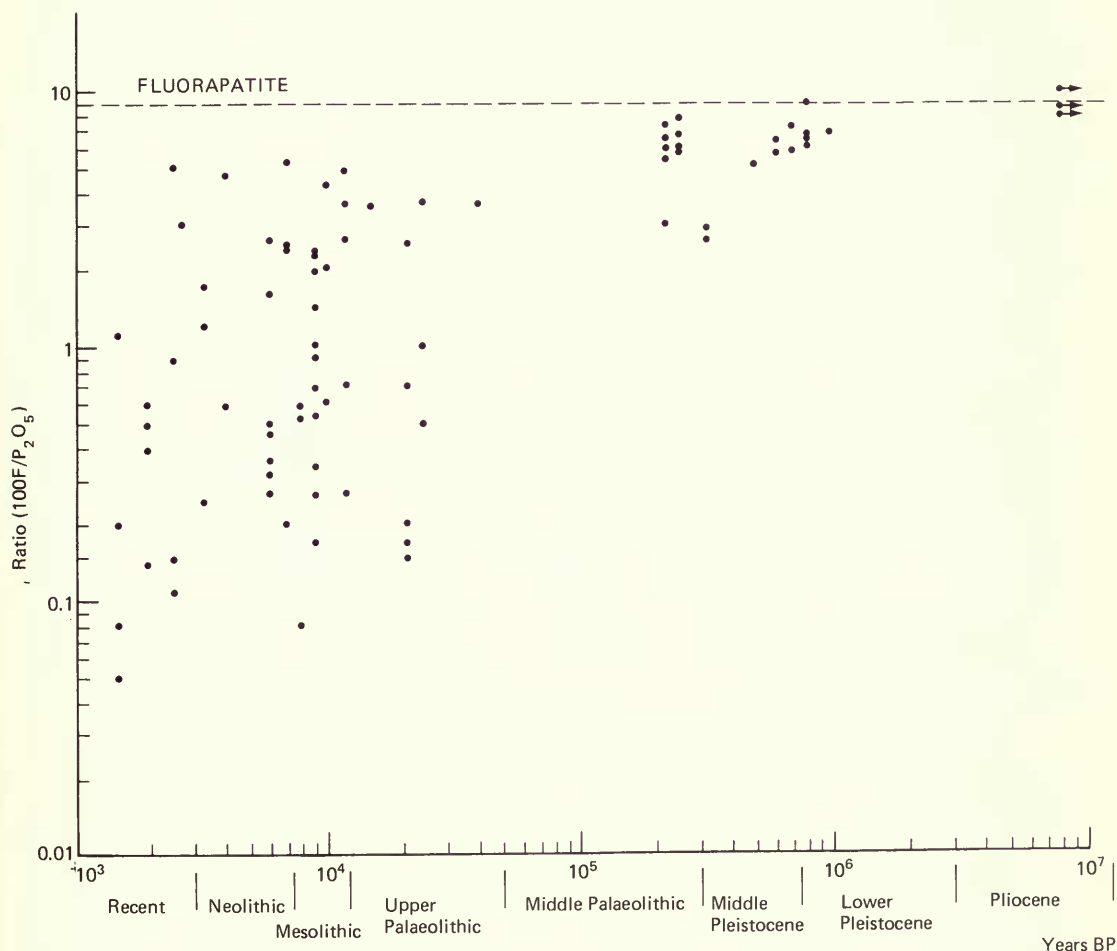

Fig. 1 Fluorine/phosphate ratios in fossil skeletal samples from the British Isles (including Ireland but excluding Channel Islands), plotted against their stratigraphical ages. Note the trend with increasing age towards the theoretical limit: complete transformation of apatite into fluorapatite. Redrawn by G. F. Phillips to combine data presented by Glover & Phillips (1965 : 575) with further results obtained by colleagues at the Laboratory of the Government Chemist.

Fig. 2 Fluorine/phosphate ratios in fossil skeletal samples from Europe other than the British Isles, plotted against their stratigraphical ages. Compare with Fig. 1. Redrawn by G. F. Phillips to combine data presented by Glover & Phillips (1965 : 575) with further results obtained by colleagues at the Laboratory of the Government Chemist.
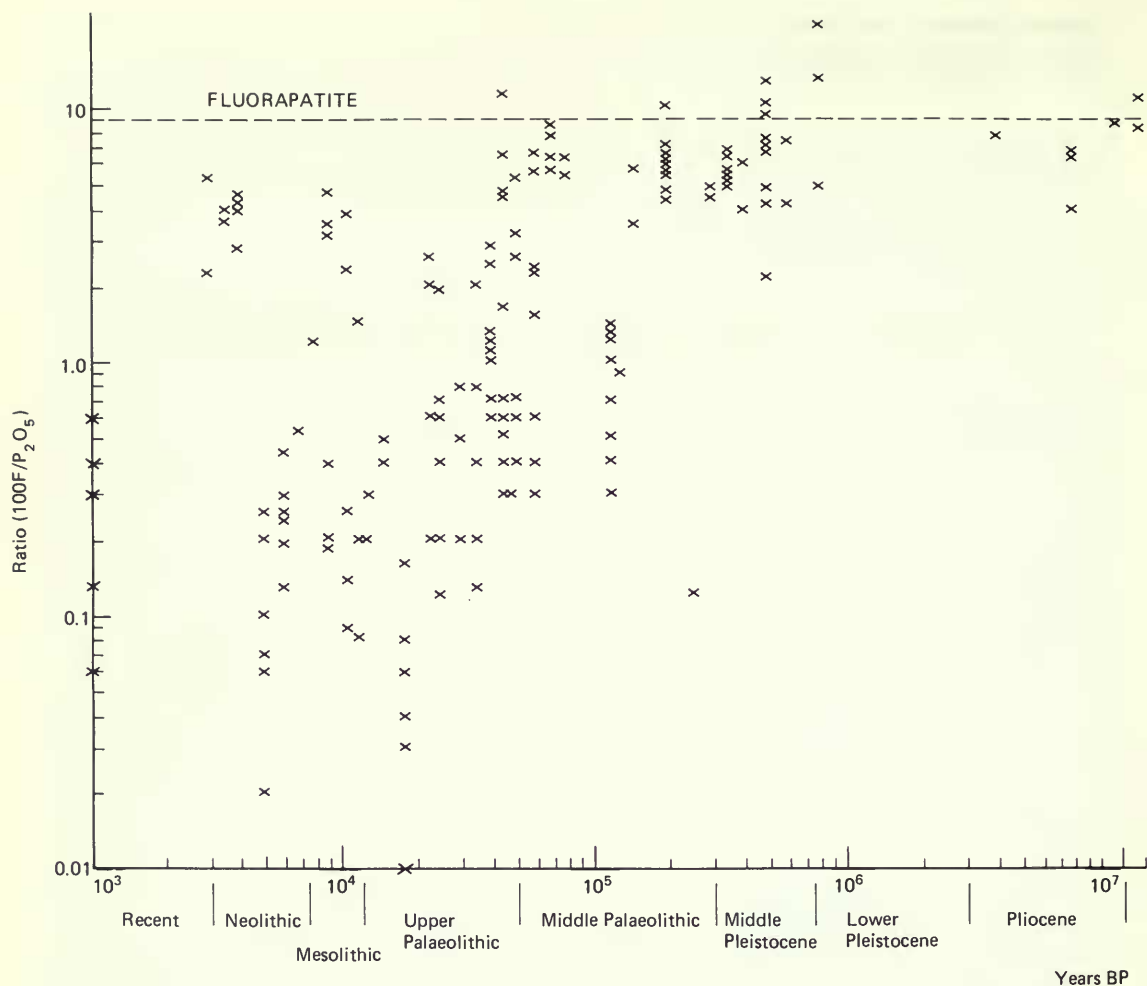
Analyses of a cross-section of a cervid antler from the Lower Gravel, Barnfield Pit, Swanscombe gave the following results:

		F %	$100F/P_2O_5$
Outer Zone	0–3 mm	2·5	7·0
Underlying layer	3–6 mm	2·3	6·3
Central cancellar tissue	6–15 mm	2·2	6·3

Glover & Phillips (1965 : 575) illustrated the relationship between the geological age and the fluorine/phosphate ratio for 345 samples of vertebrate fossils which were analysed for the BM(NH) at the Laboratory of the Government Chemist between 1955 and 1965. G. F. Phillips here re-presents the European data, with the addition of subsequent results obtained by his colleagues, as Figs 1 and 2, in which fluorine/phosphate ratio is related to stratigraphic age for material respectively from the British Isles and from mainland Europe. Although there is an upward trend in fluorine/phosphate ratio from Holocene (Recent) to Miocene, the broad spread

Table 1 Estimation of fluorine in material from Lagow, Texas (after Oakley & Howells 1961: 545). X-ray diffraction data: difference in mm between $2\theta_{(004)}$ and $2\theta_{(140)}$ for copper-Kα radiation in 19-cm diameter powder-camera.

Human rib, Lagow	3·13
Human tibia, Lagow	3·12
Mammoth ischium, Lagow	2·65
Camelops bone, Lagow	2·73
Bone from Lower Schuler Sands, condition friable	2.80
Bone from Lower Schuler Sands, condition hard	2·75
Equus bone, Hill Gravel	2·75
Bison vertebra, Upper Schuler Clay	2·71
Compare:	
Recent animal bone	3·34 (F = 0·03%)
Fossil human skull from Midland, Texas	3·13 (F = 0·70%)
Fossil human skeleton from Tepexpan, Mexico	2·67 (F = 1·50%)

of the figures indicates the danger of trying to employ the ratio as an index for geological age without taking into account local factors which in so many cases affect the degree of fluorination of vertebrate fossils.

When a long series of fossil skeletal materials from a given site require to be analysed for their fluorine content, microchemical analysis of the samples can be replaced in large measure by employing the X-ray powder diffraction technique which was introduced by Niggli, Overweel & van der Vlerk (1953). In this method the distance between a suitable pair of lines on an X-ray powder photograph of the bone apatite varies with the fluorine content. In order to indicate the corresponding fluorine percentage in the materials under investigation, the tabulated results should include measurements which have been made on two or three samples analysed chemically. As an illustration of an application of this technique I reproduce here (Table 1) a summary of the results obtained when the age of a fragmentary human skeleton from the Lagow Sand Pit, Texas was investigated. The X-ray diffraction data of the samples were obtained by G. F. Claringbull and the late R. J. Davis in the Department of Mineralogy, BM(NH).

The fossil animal bones from Lagow, which date from a temperate stage prior to the Last Glacial event of the Pleistocene, gave highly homogeneous results for fluorine content clustering between measurements of 2·65 and 2·80 mm for six samples of bone, while the two human bones gave readings which are clearly out of this range. But the human bones were evidently not modern, for their fluorine content indicates a slight degree of mineralization, apparently comparable with that of the Midland skull from Texas. On the other hand they appear to be much later than the fossil fauna characteristic of the formation, and tentatively they can be interpreted as probably intrusive burials dating from early Post-Pleistocene times.

When Carnot's fluorine dating method was first used, and when it was reintroduced (Oakley 1948, 1951, Oakley & Montagu 1949, Oakley & Hoskins 1950), no use was made of nitrogen analysis and uranium analysis for relative dating. When the laboratory investigations of the Piltdown fossils were undertaken (Weiner, Oakley & Le Gros Clark 1953, Oakley *in* Weiner *et al.* 1955 : 247–253 & 254–261) it was found that the most effective method of relative dating of fossil bones preserved under temperate conditions was to analyse the skeletal material for fluorine, 'uranium' and nitrogen. It is relevant to mention here that the Lagow investigation (Oakley & Howells 1961) utilized the combined techniques of fluorine, 'uranium' and nitrogen dating, but only the fluorine results are quoted here.

A few of the factors which limit the usefulness of fluorine dating should be mentioned. As already indicated (p. 3), it is unreliable in tropical regions and or where the soils are rich in volcanic minerals. Since the adsorbtion of fluorine by buried bones depends on the percolation of fluorine-bearing ground-water, the fluorine dating method also gives unreliable results in exceptionally arid regions (e.g. Transvaal cave deposits) and in calcareous cave deposits where layers or seams of calcite commonly prevent the free circulation of the ground-water.

The question of what time-interval is adequate to permit differentiation by fluorine analysis can best be answered in the light of experience at a particular locality. The amount of fluorine that has accumulated in the bones or teeth during Post-Pleistocene periods in Britain, at any rate, is usually quite inadequate for differentiating them very clearly. Thus the range of fluorine content in a series of Neolithic bones would overlap that of Bronze Age bones excavated in the same region. On the other hand Pleistocene bones, and bones buried at the same locality only a century or so ago, would in both cases be clearly separable from Neolithic or Bronze Age groups by fluorine analysis. From the point of view of the present paper it is worth quoting figures obtained when two groups of Upper Pleistocene bones were analysed from a site in Austria (Kulna cave, specimens collected by K. Velloch):

	$100F/P_2O_5$
Bones (3) from Magdalenian layer	0·4–0·5
Bones (9) from Mousterian layer	0·3–1·4

From these results it is evident, as would be expected, that on average there is a greater concentration of fluorine in the Mousterian bones. However, as the ranges of fluorine for the Magdalenian bones falls within that of the Mousterian, it would not be possible to use fluorine-dating to decide whether a bone of uncertain horizon in this cave, with a ratio of say 0·4, had been derived from the Magdalenian deposits or from the Mousterian deposits at this site. Even at such a site it would nevertheless be valuable to determine the fluorine content (or better the ratio $100F/P_2O_5$) in a specimen of uncertain horizon because the results of the analysis would indicate the degree of probability that the specimen had been derived from the younger or from the older layer.

At a given level of fluorination within a bone-bearing deposit, the fluorine content of a specimen may prove to vary with the texture or the type of vertebrate tissue that was sampled. It is therefore essential in fluorine dating (as in all methods of relative dating by analysis) to compare like with like as far as possible. Fluorine is adsorbed almost uniformly throughout the thickness of compact bone, compact antler or dentine (e.g. elephant tusk or root of tooth), and at about the same rate in all these materials; but it is adsorbed at slightly higher rates in spongy bone or antler, while enamel is a law unto itself and is better excluded from comparative series when other dental material is available. Cancellar or spongy tissue is liable to be contaminated by silt or other mineral matter, but if comparison is made between fluorine/phosphate ratios rather than between the actual fluorine contents of the samples this complicating factor is eliminated.

References. Publications on fluorine in fossil bones and teeth, including applications of the 'fluorine test' for relative antiquity of fossil vertebrate remains: Bergman & Karsten 1952, Bromehead 1943, Carnot 1892a, b, 1893, Cook 1960, Day & Molleson 1976, Földvari-Vogl & Kretzoi 1961, Glover & Phillips 1965, Hoskins & Fryd 1955, Mehta & Simpson 1975, Middleton 1844, Milton, Lidell & Chivers 1947, Oakley 1948, 1950, 1951, 1954a, b, 1955b, c, 1963c, 1969, 1974, Oakley & Gardiner 1964, Oakley & Hoskins 1950, 1951, Olsen 1950, Stewart 1951, Van der Vlerk 1957, Vayson de Pradenne 1932, Wilson 1902.

Relative dating by uranium

Uranium is adsorbed from ground-water by the apatite of bones and teeth, which accumulate this element in course of time, and thus provides a useful alternative to fluorine as an indicator of the relative ages (or of the sources) of buried skeletal remains, particularly in sands and gravels. The rate at which the uranium is accumulated in bone or tooth depends largely on the abundance of this element in the environment, but also to a considerable extent on the hydrological conditions prevailing at the site and on the nature of the mineral matrix. In clays and limestones the circulation of uranium is considerably inhibited, but in sands and gravels the accumulation of uranium and its daughter elements in skeletal material provides a most useful means of distinguishing between indigenous and intrusive specimens. 'Intruders' may of course be younger or older than the indigenous series.

The 'uranium' content of a specimen (and by 'uranium' in this context we mean all elements of the uranium family) can be conveniently assessed by a physical method: the so-called radiometric assay, which takes the form of exposing a sample of the bone, antler or tooth to a Geiger counter screened in a lead chamber, and counting its β-radiations per minute, with due allowance for any background radiation. The counts per minute due to the skeletal sample are expressed as equivalent to so many units of uranium oxide per million (e U_3O_8 ppm).

Radiometric assays are of little or no use for the relative dating of bones from post-Glacial deposits owing to the initially slow build-up of uranium, but as the adsorbed uranium generates a series of unstable daughter elements, there is a steep rise in the radioactivity of buried bones in the course of tens of millennia, so that radiometric assays usually distinguish quite clearly between fossil and recently-intruded bones in Pleistocene gravels and sands.

Radiometric assays of the 'Piltdown assemblage' showed (as fluorine had done) that it was not uniform in geological age or origin. The radiometric assay went further than fluorine analysis, for it revealed that some of the specimens (fragments of molar tooth or teeth of *Elephas* cf. *planifrons*) were more radioactive than any Pleistocene fossils known in Britain and had probably been derived from a foreign source (see p. 13).

The radiometric assay has an advantage over fluorine analysis for relative dating, since it can be made without any destruction of material if the bone or tooth can be placed in a space measuring $4 \times 3 \times 2$ cm. Its surface can then be assayed directly. In the case of larger specimens the assay can be made on about a gram of bone or dentine powder drilled out by means of a dental burr.

It should be noted that the weak radioactivity due to carbon-14 (^{14}C) atoms in the residual organic matter or protein of bone or dentine is never confused in practice with the much stronger radioactivity of the adsorbed uranium in the apatite of fossil bone, dentine or enamel. This is evident enough from the fact that fresh bone with maximum content of ^{14}C shows a *nil* count rate on a radiometric assay devised for measuring the uranium content of the specimen.

References. Publications on uranium in fossil bones and teeth and on uses of the radiometric assay in determining the relative antiquity of vertebrate specimens: Bowie & Davidson 1954, 1955, Davidson 1953, Fleischer, Price & Walker 1965, Howell *et al.* 1972, Molleson & Oakley 1966, Oakley 1961, Oakley & Rixon 1958.

Relative dating by nitrogen

The nitrogen content of bones and teeth is due to the protein collagen which forms the organic matrix on which the phosphatic mineral matter has been deposited in the course of formation within the body.

Electron microscopy has made it possible to study the fibrous structure of collagen. In 1955 J. T. Randall and A. V. W. Martin prepared an electron micrograph of the decalcified residue of a sample of the Piltdown mandibular bone, revealing well-preserved collagen fibrils with characteristic banding at intervals of 640Å (Oakley 1955c: pl. 30, fig. 11). Residue of a decalcified sample of the Lloyd's site woolly rhinoceros bone, although about 30 000 years old, proved to contain intact collagen fibrils, but they were partly denatured and showed only vague shadows of the original banding (Fig. 5, p. 21). The only fossils in which collagen fibrils had been found previously were samples of mammoth ivory from frozen ground (Figs 3, 4; see also Randall *et al.* 1952).

It is noteworthy that when Randall & Martin (*in* Oakley 1955c : 255) undertook to examine the collagen in samples of the teeth in the Piltdown mandible, the first results were inconclusive for in drilling the samples frictional heat had denatured the collagen; this takes place at 70–100°C.

On chemical analysis fresh bone or dentine usually proves to contain 4–5% nitrogen derived from the collagen matrix. After burial the collagen in the bones and teeth is gradually lost but at a relatively slow, sometimes almost uniformly declining, rate. The rate of the regression of nitrogen in buried bone and dentine depends on the physical, chemical and bacteriological conditions of the immediate environment. Nitrogen is lost most rapidly under oxidizing conditions. Under
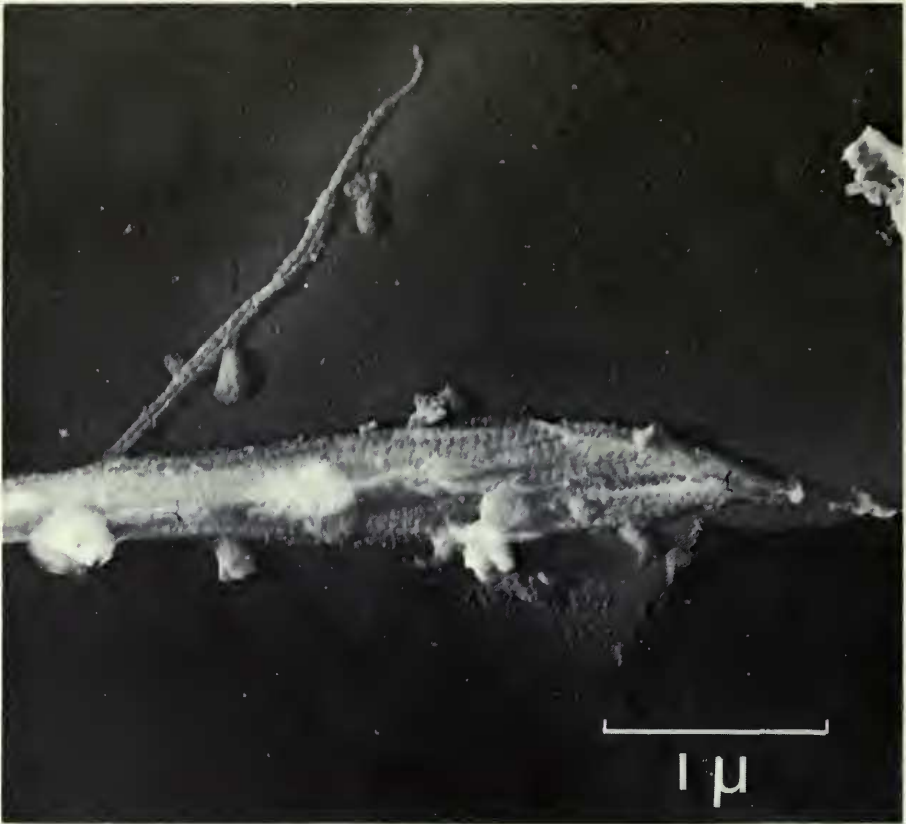
Fig. 3   Electron micrograph of decalcified residue of mammoth ivory from permafrost, Alaska,
     showing banded collagen fibrils. Shadowed with palladium and gold. × 30 000 (approx.) Photo:
     C. G. Ogden.

some circumstances the extent to which the nitrogen content has regressed provides a most useful
guide to the antiquity of a bone or tooth. Nitrogen analysis is notably useful for the relative dating
of bones and teeth when they are of several different geological ages but preserved together under
identical conditions. Relative dating by nitrogen content is particularly useful in cases where the
bones or teeth are too recent to be within range of the fluorine or uranium dating methods. As with
fluorine, it may be unreliable in limestone cave deposits where the vertebrate items have been sealed
by films of calcite, but it gives good results in permeable deposits on open sites. It is always import-
ant that the control series should include a sufficient number of samples to indicate the range of
variation of nitrogen content at a given horizon, and also that allowance should be made for
variation in the type of material tested (e.g. outer layer of compact bone, spongy bone, dentine,
enamel). The principle that like must be compared with like is particularly important in relative
dating by nitrogen content. The nitrogen test is perhaps most valuable as a means of cross-
checking the results of uranium or fluorine analysis of bone believed to be of Pleistocene age on
open sites.

   Nitrogen analysis of *unburnt* bones from archaeological sites is now frequently used as an
indirect, but very convenient guide as to whether a specimen contains enough residual protein
for ^{14}C dating and if so, what quantity of material will be required. The carbon/nitrogen ratio
in bone has been given as 3 : 1 (Orr & Berger 1965), but in practice I have found it approximates
more closely to 2·5 : 1. Thus, if a bone has had its collagen content reduced in course of natural
weathering to the level of 1 % N, about 100 grams of this bone would be needed to yield 2·5 grams
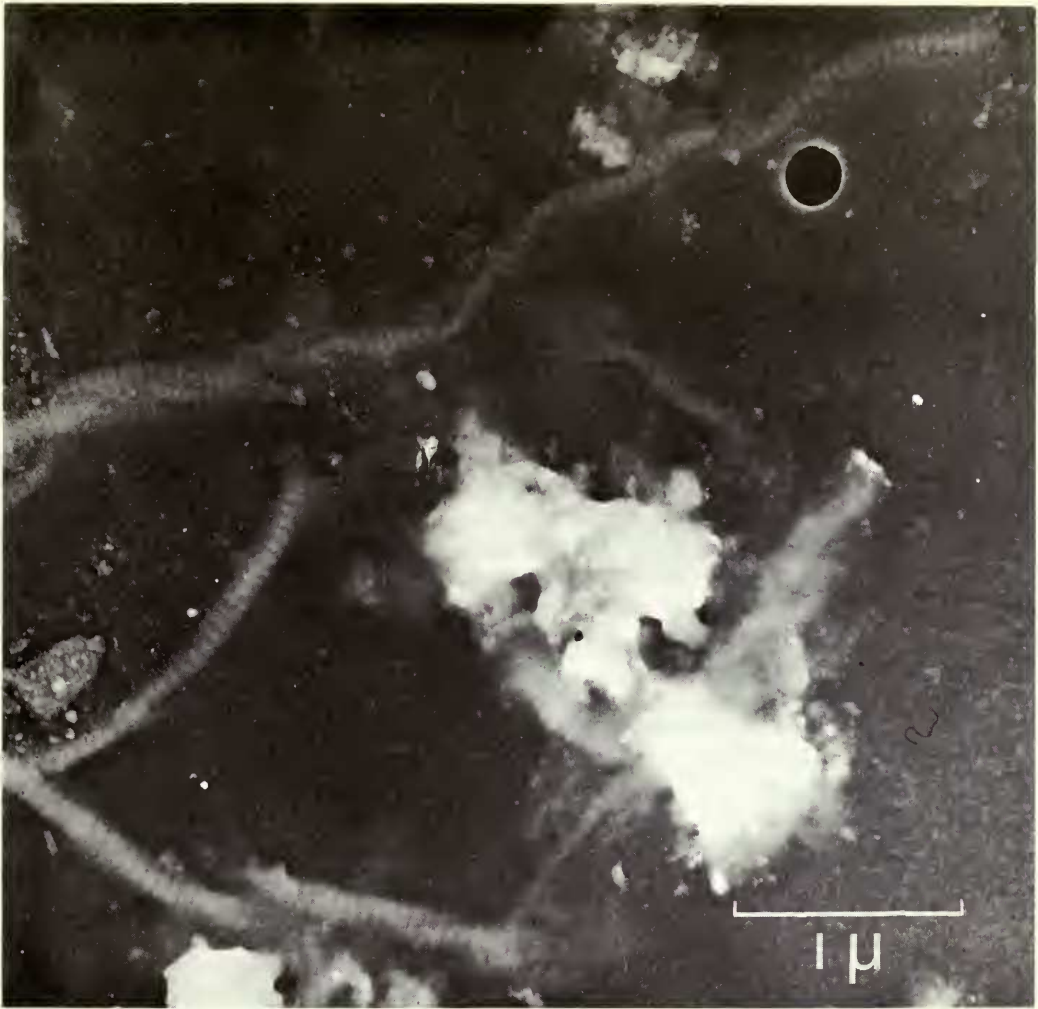of carbon.

Fig. 4 Electron micrograph of decalcified residue of mammoth ivory showing banded collagen fibrils. Shadowed with palladium and gold. × 30 000 (approx.). Photo: C. G. Ogden.

Nitrogen in bone, antler, dentine or enamel was at one time usually determined by the chemical micro-Kjeldahl method, but this is only applicable with accuracy to samples weighing more than 10 mg. Most of the nitrogen determinations reported in this paper were made by a method of microcombustion (*fide* F. B. Strauss). The amino-acid or protein content of skeletal materials can be measured by chromatography (p. 20). It is of course essential to be certain that samples sent for analysis in 'nitrogen dating' are free from contamination by any nitrogenous hardening agent, adhesive or moulding medium (e.g. size, celluloid, glue or gelatin). Whenever possible only *untreated* bone or dentine should be submitted. If there is any doubt about this, as there often is when the specimens are from an old museum collection, the analyst should be warned so that he can, as a precaution, wash the ground or drilled samples in warm water (< 70°C) and in acetone or other solvents capable of removing nitrogenous contaminants but leaving any original collagen intact. All the samples in a comparative series should receive the same treatment.

As bone protein or collagen decays in the course of fossilization it is broken down into the various component amino-acids, which are leached out or retained for varying lengths of time depending on the local conditions (Abelson 1956). Some amino-acids were found surviving even in the bones of fishes embedded in hard shale at least 350 million years old in the Devonian of

Ohio. To assess the degree of degradation of collagen in fossil bone a sample is first dissolved in a suitable acid, and the amino-acids present in the resulting hydrolysate are then determined by means of chromatography. A paper-chromatogram (Fig. 6, p. 21) prepared from a sample of the ulna of a woolly rhinoceros preserved in Upper Pleistocene clay at the Lloyd's site, London, showed strongly the main amino-acids composing collagen. In marked contrast the Galley Hill skeleton (p. 17), although only dating from the Bronze Age but preserved in a gravelly matrix, gave a chromatogram which showed only a few of these amino-acids and those in reduced strength. It is impossible to use the residual amino-acids in fossil bones determined by chromatography as a method of relative dating, but as they are subject to a process of change known as racemization[2] they do provide a method of chronometric dating (Bada & Protsch 1973, Bada *et al.* 1974, Fleming 1976 : 193–200).

The concentration of nitrogen does not decrease with absolute uniformity in time because it is influenced by the variety of factors listed above. Many more observations are required before we shall know the extent to which climatic factors govern the rate of decline in the nitrogen content of fossil bones; work along these lines has already been published (Buczko & Vas 1977, Buczko 1978, Vonach 1976). Measurements on buried bones on the Hungarian Plain indicated that the minima in nitrogen content coincide fairly closely with the climatic maxima during the last 9 000 years. The effect of temperature on protein decay in bone has been studied experimentally (Ortner, von Endt & Robinson 1972). Further light might be thrown on this problem by analysing pieces of bone buried under various microclimates and determining their nitrogen contents after the lapse of various short periods of time.

References. Publications on the collagen, nitrogen and amino-acid contents of fossil bones and teeth: Abelson 1956, Ascenzi 1955, 1969, Bada & Protsch 1973, Bada *et al.* 1974, Buczko & Vas 1977, Cook 1960, Cook & Heizer 1947, Ezra & Cook 1957, Fleming 1976 (esp. 193–200), Garlick 1969, Isaacs, Little, Curry & Tarlo 1963, King & Bada 1979, Oakley 1954a, 1955c, 1963b, Orr & Berger 1965, Randall *et al.* 1952, Vonach 1976, Weiner, Oakley & Le Gros Clark 1953 (esp. 143–144).

Uses of combined techniques

Fluorine, uranium and nitrogen analyses have been used in combination mainly for relative dating of fossil bones and teeth (Day & Molleson 1973 : 146, 148, Molleson & Oakley 1966, Oakley 1955a, b, c, 1963c, 1969). In addition to many applications to fossil remains in Europe, the combined techniques have been applied to remains from hominid sites in Asia (Day & Molleson 1973, Oakley 1969 : 44–45), Africa (Oakley 1957, 1958c) and U.S.A. (Oakley & Howells 1961).

Analyses of vertebrate specimens for fluorine, uranium and nitrogen have sometimes proved valuable in helping to establish the origins of specimens of doubtful provenance. A notable example is a molar tooth of *Hippopotamus major*, BM(NH) 18304, in MacEnery's collection from Kent's Cavern, a specimen which a number of workers suspected had really originated in Villafranchian deposits of the Mediterranean region. On analysis of this tooth the fluorine/phosphate ratio proved to be 5·7, compared with 5·3 in a tooth from the Val d'Arno, Italy, whereas the fluorine/phosphate ratio in teeth from Kent's Cavern has not been recorded as exceeding 0·6 (Oakley 1964b). These results strongly suggest that it *had* been imported from the Villafranchian deposits of southern Europe and accidently incorporated in the Kent's Cavern collection. Other instances where analyses of fluorine and uranium in fossil bones have proved useful in indicating the provenance of the specimens are worth quoting here.

The 'Piltdown I assemblage' included a molar tooth of *Anancus* cf. *arvernensis*. On analysis its enamel showed e U_3O_8 97 ppm and the fluorine/phosphate ratio proved to be 6·4. The enamel

[2]There are two distinct molecular forms of amino-acids, known as optical isomers, and these are distinguished as L or D forms according to whether they rotate the plane of polarization of polarized light to the left (laevorotatory) or to the right (dextrorotatory). In living tissue proteins are composed almost entirely of L amino-acids, but after death the amino-acids slowly racemize, that is to say L forms are converted into D forms, the proportions of the two tending to become equal. Fossil bones are thus found to contain both D and L forms and the ratio of D to L increases with the advancing age of the specimen. The relative amounts can easily be measured by an optical method. Aspartic acid and iso-leucine are the amino-acids most commonly used in this method.

of a typical tooth of *Anancus arvernensis* from the Red Crag of Suffolk showed e U_3O_8 38 ppm and a fluorine/phosphate ratio of 7·0. Since e U_3O_8 is recorded as ranging from 13 to 174 ppm in teeth from the Red Crag of Suffolk, the disparity between the uranium content of the enamel of the *Anancus* tooth used for comparison and the one in the 'Piltdown' group is insignificant. However, the enamel of another 'Piltdown' tooth, of obviously extraneous origin and identified as *Elephas* cf. *planifrons*, contained e U_3O_8 520 ppm and the cementum of the same tooth e U_3O_8 610 ppm[3].

When the latter radiometric assays were compared with those of teeth of the *Elephas planifrons* group from numerous localities in Europe, Asia and Africa the only ones which were closely comparable were from Ichkeul in Tunisia. The dentine of a typical molar tooth from there showed e U_3O_8 580 ppm and the enamel e U_3O_8 480 ppm. But the fact that Ichkeul was unknown as a source of Lower Pleistocene fossils until 1949 greatly reduces the likelihood that the 'Piltdown' elephant molar fragments originated there. Dr Vincent Maglio has informed me, however, that specimens of *Elephas* cf. *planifrons* (now *Archidiskodon africanavus*) have also been found at six other north African sites (a second site in Tunisia, three in Chad and two in Morocco). He agreed that 'the most likely source for the Piltdown fragments is north Africa'. It is surely significant that uranium fields occur in Morocco and in Tunisia.

A hominid femur, OH34, was excavated in 1962 from Olduvai Gorge Bed III at site JK2. On account of its strange preservation it was thought wise to analyse samples of this specimen in an attempt to confirm its contemporaneity with other fossils undoubtedly from Olduvai Bed III. The level of 'uranium' proved to fall within the range recorded in other bones from Bed III, and the fluorine content was consistent with its recovery from this deposit (Day & Molleson 1976).

The usefulness of the combined techniques is also illustrated by the results obtained through applying them to the problem of the age and origin of the remains of sabre-tooth cats reported from British cave deposits. In 1876 a canine tooth of *Homotherium* ['*Machairodus*'] was found in an Upper Palaeolithic layer in Robin Hood's cave, Creswell Crags, Derbyshire (Dawkins 1876). Some authorities doubted whether this was a genuine find, suspecting that it had been fraudently placed at the site, and it had really originated in alluvial deposits of Early Pleistocene age in France or Italy, where specimens of this genus are not uncommon. Comparison of the fluorine, 'uranium' and nitrogen contents of the Creswell tooth with Villafranchian specimens from the main Continental localities dispels this suggestion (Table 2), but other possibilities have to be considered. Was the Creswell canine derived from a Lower Pleistocene deposit in Derbyshire and brought into the Robin Hood's cave by prehistoric man? There are many instances of fossils having been treasured by stone age man and transported to a considerable distance from their natural source. *Homotherium* canines, but specifically different from the Creswell specimen, have been found in association with Lower Pleistocene fossils in a fissure deposit at Doveholes, also in Derbyshire. Yet in their lack of nitrogen, high fluorine content and high 'uranium' content they contrast sharply with the Creswell specimens whose composition on the other hand agrees closely with that of local Upper Pleistocene cave mammal remains.

Table 2   Fluorine, nitrogen and uranium content of *Homotherium* teeth from Creswell Crags and other European sites.

Source of *Homotherium* dentine tested	Nitrogen %	Fluorine %	$100F/P_2O_5$	Uranium e U_3O_8 ppm
Val d'Arno, Italy	0·2	1·6	5·3	35
Mt Perrier, France	nil	1·9	6·3	30
Doveholes, Derbyshire	nil	2·5	8·6	68
Creswell, Derbyshire	2·1	0·2	0·8	< 1
Kent's Cavern, Devon	1·2	< 0·01	< 0·1	5

[3]Fluorimetric determination of uranium in this tooth indicated U_3O_8 = 1000 ppm.

Table 3   Fluorine and nitrogen content of mammalian teeth from Kent's Cavern.

Specimens of dentine	Nitrogen %	$100F/P_2O_5$
Homotherium canine, BM(NH) 14954, from cave-earth	1·2[4]	<0·1
Mammuthus molar from cave-earth	0·8	<0·1
Ursus molar from hard breccia	0·2	<0·1

If it were still to be maintained that the Creswell specimen had been fraudulently planted, its composition could only be accounted for by supposing that it had been obtained from some other similar limestone *cave* deposit elsewhere. For in my experience it is only in limestone cave deposits that Pleistocene vertebrate specimens are so deficient in fluorine as this one. Teeth of *Homotherium* have in fact been reported from Upper Pleistocene cave-earth in Kent's Cavern, Torquay. It has been widely assumed by vertebrate palaeontologists that these specimens were residues from some much older deposits in the Torquay Cave system. The composition of one of the Kent's Cavern canines was therefore tested. The fluorine content of the dentine proved to be negligible and the 'uranium' content to be very low. The failure of these two elements to circulate in calcareous cave deposits is recognized, but if these specimens were derivatives considerably older than the Upper Pleistocene mammoth tooth that apparently occurred in the same cave-earth, they should contain substantially less nitrogen. In fact they proved to contain just as much (Table 3). Thus it can be inferred provisionally that sabre-tooth cats (*Homotherium*) survived as rarities in Britain during Upper Pleistocene times, and that the last examples were contemporary with Middle Palaeolithic (Mousterian) and possibly with the earliest Upper Palaeolithic men. The survival of *Homotherium* on the Continent into late Middle Pleistocene times is already well established (Adam 1961).

References: Adam 1961, Dawkins 1876, Day & Molleson 1973, 1976, Molleson & Oakley 1966, Oakley 1955a, b, c, 1957, 1958a, c, 1963c, 1964b, 1969, Oakley & Howells 1961.

European fossil hominids to which analytical methods of relative dating have been applied

Note on the Tables

The tables show significant analyses of fossil hominids of Europe compared with similar analyses of associated mammalian faunas. No authentic fossil hominid has been reported from Lower Pleistocene deposits in Europe. Table 4 (p. 25) shows the composition of mammalian teeth identified as derived from the Lower Pleistocene (Red Crag) of Suffolk but which were used to form part of the fraudulent faunal assemblage placed in association with the forged hominids Piltdown I and II, Sussex. The fluorine/phosphate ratios and the uranium contents of these Lower Pleistocene fossils range to higher levels than in Middle Pleistocene material from southern Britain. (See Table 5, p. 27).

Between 1948–75, fluorine, phosphate, iron and carbonate determinations (and occasionally uranium, manganese, sodium, potassium and chloride) were made in the Laboratory of the Government Chemist, London, by R. H. Settle and E.C.W. Maycock (1948–49), Dr C. R. Hoskins (1949–53), C. F. M. Fryd (1953–57), P. J. Hardwick (1953–63), A. D. Baynes-Cope (1954–58), Dr J. R. Cooke (1958–59), A. A. Christie (1959), G. F. Phillips (1960–66), M. J. Glover (1963–65), J. Roburn (1966-67), E. C. Hunt (1968–70) and N. M. Soutar (1971–75). A few of the nitrogen determinations were made in the same Laboratory by E. I. Johnson (1957–61), P. J. Cooper (1965) and N. M. Soutar (1971–72), but the majority were made by Dr G. Weiler (from 1953) and Dr F. B. Strauss (1953–77) in the Microanalytical Laboratory, Oxford. The calcium carbonate was estimated in the Laboratory of the Government Chemist on the basis of determination of CO_2. The uranium determinations expressed as U parts per million were made by Dr R. L. Fleischer in the General Electric Research Laboratory, Schenectady, New York, using the fission-track method; the larger number expressed as $e\ U_3O_8$ were made on the basis of radioactivity measurements by

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[4]Washing the sample in warm water and then in acetone to remove any possible traces of nitrogenous preservative effected no reduction in the nitrogen content.

S. H. U. Bowie and Dr C. F. Davidson, Atomic Energy Division, Institute of Geological Sciences (1953–54) and by twelve members of staff of the Department of Palaeontology (including the Subdepartment of Anthropology), BM(NH), 1955–75.

The majority of the tables plot the percentages of the following components: F, P_2O_5, $CaCO_3$, N (w = after washing) and the uranium content expressed as U or e U_3O_8 in parts per million (ppm). The ratio $100F/P_2O_5$ is also indicated. Radiocarbon (^{14}C) dates are designated by bc or bp (before present); calendar dates, derived by correction of the radiocarbon dates (Clark 1975) are designated by BP or BC. In Tables 6 and 17 some radiocarbon ages (bp) of hominid bones are given, and in Tables 6, 9 and 17 the radiocarbon ages (bp) of associated mammalian remains are shown. References to the radiocarbon reports are given at the end of the descriptions of the sites. CFH No. – Catalogue of Fossil Hominids **2** (Oakley, Campbell & Molleson 1971).

British Isles

AVELINE'S HOLE (Somerset). Skeletal remains of *Homo sapiens* generally attributed to the final stage of the British Upper Palaeolithic have been recovered from Aveline's Hole, a blocked cave on the east side of Burrington Combe in the Mendip Hills. They include an adult male calvaria encrusted with stalagmite, discovered by W. Buckland in 1823 (Aveline's Hole 1), a mandible excavated by R. Bright before 1840 (Aveline's Hole 2) and a cranium with mandible obtained during excavation by the Bristol Speleological Research Society in 1914 (Aveline's Hole 9).

Aveline's Hole 1: F = 0·47%, $100F/P_2O_5$ = 1·98, e U_3O_8 = 2 ppm, N = 3·8% (w)
Aveline's Hole 2: F = 0·29%, $100F/P_2O_5$ = 1·04, e U_3O_8 = nil, N = 1·8% (w)
Aveline's Hole 9: F = 0·48%, $100F/P_2O_5$ = 2·28, N = 3·47%.
Human metacarpal, Aveline's Hole 1914 unnumbered: F = 0·20%, $100F/P_2O_5$ = 0·92, N = 3·49%
Mammalian bones from cave-earth, Aveline's Hole: F = 0·07%, $100F/P_2O_5$ = 0·26; F = 0·20%, $100F/P_2O_5$ = 0·67; N = 1·4–3·0%
See Table 6, p. 28.

The ^{14}C dating of residual collagen in a human femur (1914 unnumbered) from Aveline's Hole was determined in the British Museum Research Laboratory as 9114 ± 110 bp (BM-471). This result compares significantly with the ^{14}C dating of stalagmite filling the Aveline's Hole 1 skull: 8100 ± 150 bp (GrN-5393), a determination kindly made by Dr J. C. Lerman in the Groningen Radiocarbon Laboratory (information *in lit.*, 1968). Some of the Aveline's Hole skeletal material is therefore as late as Lower Flandrian, but the total amount (including fragments of *c.* 50 skeletons found in 1797 and subsequently lost) is sufficient to make it probable that occupation or use of the cave began in Devensian times.

References: Barker, Burleigh & Meeks 1971 : 179–180; Campbell 1977 : 163, Table 4; Molleson 1977 : 88, 90; Oakley 1971*a* : 17–19; Tratman 1977.

BADGER HOLE (Somerset). A child's mandible (Badger Hole 1) and pieces of an adult cranium (Badger Hole 3) were found in hard cave-breccia at this site by H. E. Balch in 1939 and 1945.

Badger Hole 1: N = 1·58%
Badger Hole 3: N = 0·93%
Associated *Crocuta* bone N = 2·5%
See Table 6, p. 28.

The ^{14}C dating of charred bone fragments from layer 1 (Proto-Solutrean): > 18 000 bp (BM-497). It is inferred that the Badger Hole skeletal material is stratigraphically Middle Devensian.

References: Barker, Burleigh & Meeks 1971 : 168, Campbell 1977 : 51, 88, Molleson 1977 : 87, 88, Oakley 1971*a* : 19–20.

BAKER'S HOLE (Kent). In 1903 G. White described the finding in the previous year of a human skull in brickearth at Baker's Hole, Ebbsfleet. Analyses have shown that this skull was a Post-Pleistocene intrusion.

Homo skull, Baker's Hole: F = 0·06%, 100F/P$_2$O$_5$ = 0·2, e U$_3$O$_8$ = nil, N = 2·04% (w)
Mammuthus, Baker's Hole: F = 1·2%, 100F/P$_2$O$_5$ = 4·3, e U$_3$O$_8$ = 15 ppm, N = 0·32% (w)
See Table 6, p. 28.

Reference: Oakley 1971*a* : 20.

BURY ST EDMUNDS (Suffolk): see under Westley (p. 26)

CRESWELL CRAGS (Derbyshire). Human teeth and bones from late Upper Palaeolithic (Creswellian) occupation layers in three caves in the Creswell Crags have been analysed, and compared with associated Late Glacial mammalian bones and teeth.

Mother Grundy's Parlour 1, cranium of child discovered 1876; dentine of molar: N = 0·95% (w)
Probably Mother Grundy's Parlour, *Meles* molar dentine: N = 1·20% (w)
Pin Hole Cave 1, skeleton of child from upper red cave-earth; ilium bone: F = 0·06%, 100F/P$_2$O$_5$ = 0·27, e U$_3$O$_8$ = nil, N = 3·41%
Pin Hole Cave, mammalian bone: F = 0·54%, 100F/P$_2$O$_5$ = 2·5, e U$_3$O$_8$ = nil, N = 1·94%
See Table 6, pp. 29–30, under Mother Grundy's Parlour, Pin Hole Cave and Robin Hood's Cave; also Table 2, p. 13

The composition of the ilium of Pin Hole Cave 1 compares unfavourably with that of the mammalian bone representing the Late Glacial fauna of the cave, but a longer series of comparative analyses would be needed before the antiquity of this skeleton could seriously be called in question.

In July 1969, J. B. Campbell made excavations in Robin Hood's Cave, and found a human frontal bone at a depth of 196 cm below the surface, associated with Creswellian shouldered points. Layer OB, in which the frontal bone was found, was identified by Campbell as 'undisturbed Late Glacial thermoclastic scree'. A sample of metacarpal bone of *Equus przewalskii* bulked with a sample of *Megaloceros* antler from the same layer was submitted to the British Museum Research Laboratory, where radiocarbon dating of the residual collagen gave a date of 10 390 ± 90 bp (BM-603). Pollen recovered from cavities in the *Equus* bone indicated a Late Glacial flora. On the basis of this evidence the human frontal bone (Robin Hood's Cave 1) has been accepted as dating stratigraphically from the Late Devensian. The unexpectedly high nitrogen content of this bone (3·89%) raises a slight doubt as to whether the bone was introduced into layer OB during post-glacial disturbance. It is to be regretted that samples of the *Equus* metacarpal and of the *Megaloceros* antler were not submitted for nitrogen analysis. There is a lesson to be learnt here, because clearly if these two items of Late Glacial fauna were known to be as high in nitrogen as the human frontal, all suspicion that this bone might be intrusive would vanish. There are well-authenticated Late Pleistocene bones from limestone cave deposits with equally high nitrogen content, and Robin Hood's Cave 1 is only suspect because a recently-discovered fossil human bone from the same cave proved to contain much less nitrogen (see below). During his 1969 excavations, Campbell recovered a number of other human bones from his layer E, consisting of tip-heap material from nineteenth century excavations. After a detailed study of these bones, Miss Rosemary Powers came to the conclusion that they were all parts of the skull of a single individual, the same as that represented by the frontal from layer OB (Powers & Campbell 1977, Campbell 1977 : fig. 175). All of the human bones from layer E so far analysed prove to be high in nitrogen (e.g. one of the maxillae: N = 3·81%).

R. D. S. Jenkinson, Curator at the Creswell Crags Visitor Centre, has kindly informed me about a fossil human mandible which was extracted from cave-earth in Robin Hood's Cave in 1974 by two schoolboys. It was situated *c*. 1 m below the travertine floor. In view of the importance of this discovery, the mandible was sent to the BM(NH) for reconstruction by R. J. Parsons. It was satisfactory to find that the nitrogen content (1·11%) was low enough to be consistent with the inference that the specimen was contemporaneous with the Devensian cave-earth.

References: Burleigh, Hewson & Meeks 1976 : 22 (Robin Hood's Cave); Campbell 1977 : 62–64 (Mother Grundy's Parlour), 47–48 (Pin Hole Cave), 64–69 (Robin Hood's Cave); Molleson 1977 : 87–89 (Mother Grundy's Parlour), 89, 91 (Pin Hole Cave), 89, 91 (Robin Hood's Cave); Oakley 1971*a* : 32 (Mother

Grundy's Parlour), 35–36 (Pin Hole Cave), 36–37 (Robin Hood's Cave); Powers & Campbell 1977 : 218–220 (Robin Hood's Cave).

FLINT JACK'S CAVE (Somerset). In 1893 or earlier R. Pavey recovered two human skulls from this rock-shelter in the Cheddar Gorge. Late Upper Palaeolithic (Cheddarian) artifacts were in presumed association. No fossil mammalian material has been reported from this site. The results of analysing these skulls gave figures consistent with their being of Late Devensian age.

Flint Jack's Cave 1: F $= 0.052\%$, $100F/P_2O_5 = 0.17$, N $= 1.36\%$ (w)
Flint Jack's Cave 2: F $= 0.122\%$, $100F/P_2O_5 = 0.4$, N $= 1.25\%$ (w)
See Table 6, p. 28.

References: Campbell 1977 : 158, Molleson 1977 : 88, 91, Oakley 1958*b*.

GALLEY HILL (Kent). In 1888 R. Elliott reported that workmen had found a skeleton of *Homo sapiens* at a depth of 8 ft (2·4 m) in the 100-ft terrace Acheulian gravel in a pit adjoining Galley Hill School in Swanscombe, Kent. In 1884 a similar discovery had been made in the same pit. For a number of years these two Galley Hill skeletons were regarded by Sir Arthur Keith as proof that *Homo sapiens* already existed in the earlier part of the Pleistocene period, but there remained doubt about their antiquity.

The more complete skeleton found in 1888 (Galley Hill 1) was acquired by Frank Corner, whose collection was offered for sale by his widow Mrs D. H. Pearson in 1948. The skeleton in question was eventually bought by Dr C. T. Trechmann, who presented it to the Subdepartment of Anthropology, BM(NH), where it has been registered under EM 249–262.

Application of the fluorine-dating method to Galley Hill 1 in 1948 indicated that it was an intrusive burial in the Acheulian gravels and dated from end-Pleistocene or early Holocene times. This was confirmed by measurement of the nitrogen-content of the skeleton in comparison with fossil bones contemporaneous with the gravel and from later deposits.

Galley Hill 1 femur: F $= 0.56\%$, $100F/P_2O_5 = 2.0$, N $= 1.61\%$ (w)
Galley Hill 1 humerus: N $= 2.04\%$ (w), e U$_3$O$_8$ = nil
Mammal bones from Middle Pleistocene gravels, Swanscombe: F $= 1.6$–2.4%, $100F/P_2O_5 = 5.7$–7.4, N $= <0.01$ to 0.2%
Mammal bones from Upper Pleistocene deposits, Swanscombe: F $= 1.0$–1.3%, $100F/P_2O_5 = 2.8$–4.6, N $= 0.2$–0.5%
Holocene (Anglo-Saxon) bone: F $= 0.05\%$, $100F/P_2O_5 = 0.2$, N $= 2.5\%$
See Table 5, p. 27 (Swanscombe).

A prepared sample of the humeri of Galley Hill 1 was later submitted to the Research Laboratory of the British Museum for radiocarbon dating (BM-86). The sample was decalcified in cold dilute hydrochloric acid. The resulting granular gel represented the residual collagen of the bone. After combustion, its ^{14}C content was measured and indicated a date of 3310 ± 150 yrs bp, or *c.* 3409 bp using the new half-life of radiocarbon. By reference to the bristlecone pine calibration curve (Clark 1975) this gives a calendar date of *c.* 3600 BP (*c.* 1650 BC), suggesting burial in late Neolithic or early Bronze Age times.

References: Barker & Mackey 1961 : 41, Clark 1975, Keith 1912 : 517, Montagu & Oakley 1949, Newton 1895, Oakley & Montagu 1949.

GOUGH'S CAVE (Somerset). The most important series of hominid skeletal remains of the British Upper Palaeolithic – Early Mesolithic are those found at various levels in Gough's Cave on the east side of the Cheddar Gorge, Somerset. These include the complete skeleton of 'Cheddar Man' discovered by R. C. Gough in December 1903 (Gough's Cave 1), and regarded as a burial pene-contemporaneous with the Upper Cave-earth; fragmentary skulls and other bones excavated by R. F. Parry in 1927-8, comprising the two[5] crania Gough's Cave 2 and 3 from layers 10–13, and unnumbered post-cranial fragments from layers 6–7; an adult mandible (Gough's Cave 8) found

[5]When this paper was originally drafted two other crania found by R. F. Parry in 1927–8, Gough's Cave 4 and 5, were included in the list, but according to the late Professor E. K. Tratman the precise provenance of these specimens is uncertain; Gough's Cave 4 has the appearance of being late Holocene, while the whereabouts of Gough's Cave 5 is unknown.

by R. F. Parry in 1928–9, and an adult parietal bone (Gough's Cave 7) excavated from layer 14 by a workman in 1950. Most of this material is preserved in Gough's Cave Museum, Cheddar, but the whereabouts of Gough's Cave 2 is unknown.

Analyses of Hominids from Gough's Cave:

Gough's Cave 1, talus: $F = 0.25\%$, $100F/P_2O_5 = 2.4$, e $U_3O_8 = $ nil, $N = 1.83\%$ (w), $CaCO_3 = 55\%$, organic $C = 3.7\%$

Gough's Cave 1, tibia: $N = 1.74$–3.95% (21 samples)

Gough's Cave 1, dentine (M_1): $N = 2.7\%$

Gough's Cave 3, cranium: $N = 2.9\%$

Gough's Cave 6, mandible: $F = 0.47\%$, $100F/P_2O_5 = 2.16$, e $U_3O_8 = 6$ ppm, $N = 3.02\%$, $CaCO_3 = 14.07\%$

Gough's Cave 7, parietal: $F = 0.09\%$, $100F/P_2O_5 = 1.37$, e $U_3O_8 = $ nil, $N = 0.71\%$, $CaCO_3 = 63.4\%$

Mammalian bones from the cave-earths:

Rangifer bone, above Gough's Cave 1: $F = 0.14\%$, $100F/P_2O_5 = 0.9$, e $U_3O_8 = $ nil, $N = 1.5\%$, $CaCO_3 = 28.2\%$

Ursus bone, layer 11: $N = 3.0\%$

Equus bone, layer 18: $F = 0.29\%$, $100F/P_2O_5 = 1.42$, e $U_3O_8 = 4$ ppm, $N = 2.1\%$, $CaCO_3 = 21.4\%$
See Table 6, p. 29.

Radiocarbon dating of the residual collagen in the left tibia of Gough's Cave 1 was undertaken by the Research Laboratory of the British Museum and the result was published as 9080 ± 150 bp (Barker, Burleigh & Meeks 1971 : 180). If the result had been calculated using the new half-life of radiocarbon it would be *c.* 9350 bp. It should not be assumed that the actual time of burial was very close to this date ($= c.$ 7400 bc), because a nearly correct calendar date could only be obtained after allowing for the natural radiocarbon variations that occurred over the period of time involved. Seven or eight thousand years is beyond the present backward limit of the bristle-cone pine calibration. On the basis of extrapolation from the existing calibration curve, the dating of 'Cheddar Man' is approximately 700 years too young. Assuming he was buried around 8100 BC he still postdated the Younger Dryas stage, the upper limit of which (8300 BC) has been generally adopted in Europe as the Pleistocene/Holocene boundary. Thus on the available evidence 'Cheddar Man' is of Flandrian age, but only just. He may reasonably be regarded as not differing from a typical member of the Upper Palaeolithic population of SW Britain. He was probably buried after Gough's Cave had ceased to be actively occupied (Molleson 1977). It should be remembered that the main period of occupation of this cave was during the Late Devensian, when it was used by hunting people with Creswellian culture which persisted with scarcely appreciable change into earliest Flandrian times.

From the point of view of using nitrogen-content in the relative dating of skeletal material, it is worth noting that the distribution of the collagenous residue containing this element evidently varies widely in fossilized long bones (see $\%N$ in left tibia of 'Cheddar Man', Table 6, p. 29). Even so, the ratio of the extremes of the range of nitrogen in a given specimen is rarely more than two.

References: Barker, Burleigh & Meeks 1971 : 180, Campbell 1977 : 4, 166, Cooper 1931, Davies 1904, Donovan 1955 : 76–104, Gray 1904, Molleson 1977 : 88, 91, Oakley 1971*a* : 22–25, Parry 1928, 1929, Seligman & Parsons 1914, Tratman 1975 : 7–24.

HALLING (Kent). A contracted burial was found by workmen in 1921 at a depth of about 6 ft (173–190 cm) in brickearth containing flint implements on an occupation floor regarded by the first investigator (W. H. Cook 1914) as Aurignacian. The skull was identified by A. Keith as belonging to T. H. Huxley's 'river-bed' type.

The application of relative dating tests to the skeleton of Halling Man did not give conclusive results, but very few specimens had been available for comparison. If the comparative series had been longer it is probable that the Halling skeleton would have been seen to fall well within the early Holocene range of composition.

Holocene bones:
  Bos longifrons: F = 0·09%, 100F/P$_2$O$_5$ = 0·32, e U$_3$O$_8$ = nil, N = 1·71%
  Caprid ('large sheep'): F = 0·41%, 100F/P$_2$O$_5$ = 1·6, e U$_3$O$_8$ = 2 ppm, N = 2·93% (w)

Pleistocene bones:
  Equus: F = 0·75%, 100F/P$_2$O$_5$ = 2·6, e U$_3$O$_8$ = 6 ppm, N = 1·23%
  Mammuthus: F = 1·30%, 100F/P$_2$O$_5$ = 3·9, e U$_3$O$_8$ = 4 ppm, N = 0·62%
  Halling Man, ulna: F = 0·9%, 100F/P$_2$O$_5$ = 3·0, N = 0·9% (w)
  Halling Man, femur: e U$_3$O$_8$ = 5 ppm.

A sample of the Halling femora was submitted to the Research Laboratory of the British Museum, where ^{14}C measurements on the residual collagen indicated a radiocarbon age of 4180 ± 190 bp (BM-249), or *c.* 4305 bp if the calculation is made using the new half-life of ^{14}C, that is *c.* 2355 bc. If one attempts correction of this date by reference to the bristlecone pine calibration curve, the probable calendar antiquity of the Halling skeleton appears to be very close to 3000 BC. This dating is in conformity with the results of re-examining the flint implements from the 'floor' overlying the skeleton, for it was found that they could fit into either a Late Mesolithic or a Neolithic context (G. de G. Sieveking *in lit.* 1967).

References: Barker, Burleigh & Meeks 1969 : 289, Cook 1914, Oakley 1963a, Oakley, Barker & Sieveking 1968.

KENT'S CAVERN (Devonshire). A few fragments of hominid skeletons have been found with Late Upper Palaeolithic (cf. Magdalenian) industry in the cave-earths of this cave. They include a left humerus (= Kent's Cavern 2) found probably in the first-foot level below the upper or granular stalagmite in Underhay's Gallery when W. Pengelly was digging in November 1878 on behalf of the British Association. A hominid ulna from Pengelly's excavations (specimen 16769) was not numbered in the *Catalogue of Fossil Hominids* (Oakley, Campbell & Molleson 1971). The level from which it came is unrecorded, but it is embedded in a matrix of cave-earth. Both specimens have been analysed.

  Kent's Cavern 2, humerus: F = 0·1%, 100F/P$_2$O$_5$ = 0·3, e U$_3$O$_8$ = nil, N = 1·38%
  Kent's Cavern unnumbered, ulna: F = 0·01%, 100F/P$_2$O$_5$ = 0·03, e U$_3$O$_8$ = nil, N = 2·11%
  See Table 2, p. 13, Table 3, p. 14 and Table 6, p. 29.

The fairly high nitrogen content of the ulna cannot be considered to throw doubt on its Upper Palaeolithic antiquity, because analyses of Pleistocene fauna from Kent's Cavern showed nitrogen content ranging up to 3·53%.

References: Campbell 1977 : 37–42, Campbell & Brazier *in* Campbell 1977 : 203–207, Molleson 1977 : 88–90, Oakley 1971a : 26–28.

KILGREANY (County Waterford, Republic of Ireland). Two human skeletons were found in a discontinuous layer of stalagmite in Kilgreany Cave during excavations in 1928 under the leadership of E. K. Tratman. The stalagmite contained remains of a Late Glacial fauna, and at first it was suggested that the human remains were contemporaneous. Further excavations in 1934 showed that the deposits were in a confused state. The Late Glacial faunal material was intermingled with bones of domesticated animals and sherds of Windmill Hill (Neolithic) pottery.

  Kilgreany A, *Homo* post-cranial bone: F = 0·11%, 100F/P$_2$O$_5$ = 0·36, e U$_3$O$_8$ = nil, N = 1·07% (w)
  Megaloceras phalange: F = 0·14%, 100F/P$_2$O$_5$ = 0·48, e U$_3$O$_8$ <1 ppm, N = 1·54% (w)
  Kilgreany B, *Homo* post-cranial bone: N = 0·85% (w)

Radiocarbon dating of residual collagen in a sample of the post-cranial bones of skeleton A gave 4580 ± 150 bp (BM-135), indicating that it was probably Neolithic, presumably a burial. Radiocarbon dating of collagenous residue in a sample of post-cranial bone of skeleton B at first proved unreliable owing to the impregnation of the skeleton with a waxy preservative. It is intended that, after more thorough pretreatment, a second attempt will be made to date this material.

References: Barker & Mackey 1968 : 4, Oakley 1971a : 28–29, Molleson & Vogel in prep.

LANGWITH CAVE (Derbyshire). In 1909 E. H. Mullins, while excavating this cave on the north side of Poulter Valley, Langwith Basset, found a human cranium together with vertebrae and phalanges representing a burial. The cave-earth in which the bones were discovered contained hearths and an Upper Palaeolithic (Creswellian) industry.

Cranium, Langwith Cave 1: N = 2·7%
See Table 6, p. 29.

The high nitrogen content of this Upper Palaeolithic skull is an illustration of the unreliability of collagenous residues for relative dating of bones in limestone cave deposits. If the bone became sealed off from percolating water by calcitic films, the decay of collagen and the leaching out of amino-acids would have been arrested at that point. Another bone of the same age in a situation where percolation of water had been unimpeded would show substantially less nitrogen representing residual collagen.

References: Campbell 1977 : 158, Molleson 1977 : 89, Oakley 1971 : 29–30.

LLOYD'S SITE (City of London). As reported by Warren R. Dawson in October 1925, workmen collected a human calvaria from redeposited London Clay 42·6 ft (*c.* 13 m) below the surface in excavations for the Lloyd's Building in Leadenhall Street. Assuming that the containing deposit formed the base of the Upper Flood-plain Terrace of the Thames, many authorities assumed that this London skull was of Upper Pleistocene age, and since it clearly belonged to *Homo sapiens* it was regarded as possibly of considerable importance. Analytical dating techniques when applied to the skull in comparison with fossil mammalian bones from the same site showed that it was probably intrusive. The chemical composition of the London skull agrees more closely with that of skulls of the Thames 'river-bed' series of Post-Pleistocene age.

Homo skull, Lloyd's site: F = 0·07%, $100F/P_2O_5$ = 0·23, N = 1·52%
Coelodonta ulna (M 12575), clay at *c.* 13m, Lloyd's site: F = 1·1%, $100F/P_2O_5$ = 4·2, N = 3·42%
Mammuthus femur, sand at *c.* 7m, Lloyd's site: F = 1·3%, $100F/P_2O_5$ = 4·8, N = 0·1%
Mammal bone, Holocene silts, London Docks: F = 0·2%, $100F/P_2O_5$ = 0·8.
Homo skull, 'river-bed' series, Mortlake: F = 0·32%, $100F/P_2O_5$ = 1·3, N = 5·25%
Homo skull, 'river-bed' series, Mortlake: N = 2·7%

A sample of the ulna of woolly rhinoceros (*Coelodonta*) from the Lloyd's site was submitted to the Groningen Radiocarbon Laboratory, where J. C. Vogel obtained a radiocarbon date on the residual collagen: 29 450 ± 350 bp (GrN-4630).

The reason for the preservation of so much of the protein in the Lloyd's rhinoceros bone is that it was embedded in an unoxidized clay—an environment in which collagen decays very much more slowly than in sand or gravel through which water percolates carrying with it soluble nitrogenous breakdown products of the protein. Thus, in marked contrast, a portion of mammoth femur found at the same site but in a layer of sand had lost most of its collagen and therefore showed only a small percentage of nitrogen. It is worth noting that the fluorine content of fossil bone may increase at about the same rate in sand as in clay. The fluorine content of the rhinoceros bone is almost the same as that of the mammoth bone.

As the collagen of the Lloyd's rhinoceros bone is almost undiminished in spite of its considerable antiquity we considered that it would be interesting to have a chromatogram prepared from an acid hydrolysate of the bone (Fig. 6). G. C. Ross of the Department of Zoology, BM(NH), who undertook this was able to distinguish the amino-acids alanine, arginine, beta-phenyl-alanine, cystine, glycine, hydroxyproline, leucine/iso-leucine, lysine, ornithine and proline. An EM-photograph of a decalcified sample of this bone showed that collagen fibres were preserved, but they had lost their characteristic banding, so were in a denatured condition (Fig. 5).

References: Oakley 1969 : 40, 1971*a* : 30, Smith 1925, Young 1938.

PAVILAND (West Glamorgan). W. Buckland was mainly responsible for the discovery in December 1822–January 1823 of the skeleton of *Homo sapiens* ceremonially buried in red ochre in the Paviland Cave (Goat's Hole) on the coast of the Gower Peninsula. This fossil skeleton (Paviland 1)

Fig. 5 Electron micrograph of decalcified residue of an ulna of rhinoceros (*Coelodonta antiquitatis*) from Upper Pleistocene clay, Lloyd's site, City of London, showing slightly denatured collagen fibrils. Shadowed with palladium and gold. × 28 500 (approx.). Original specimen in Department of Palaeontology (reg. no. M12575). Photo: C. G. Ogden.
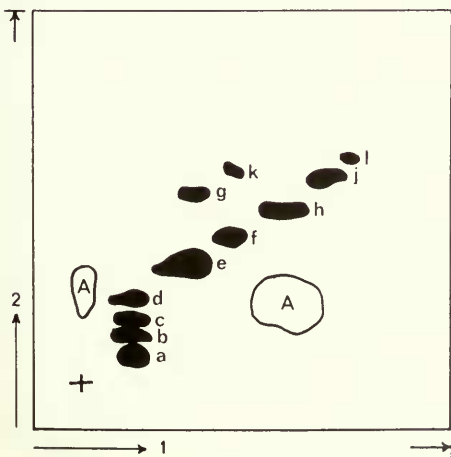

Fig. 6 Two-dimensional chromatogram, on thin-layer Silica Gel G, of amino-acids in electro-lytically desalted acid hydrolysate of rhinoceros ulna from Lloyd's site, City of London, developed in methanol : chloroform : ammonia :: 16: 16: 7 (1) and phenol (2). a, ornithine; b, cystine; c, lysine; d, arginine; e, glycine; f, alanine; g, hydroxyproline; h, valine; j, leucine/isoleucine; k, proline; l, β phenyl-alanine; A, artefact. Prepared by G. C. Ross; redrawn from tracing of chromatogram.

is that of a male aged about 25, but it became known as the 'Red Lady'. The associated fauna undoubtedly included *Mammuthus primigenius*. The skull and tusk of a mammoth had evidently been buried ceremonially with the human skeleton. Other grave-goods included an ivory bracelet, a pendant made from pulpstone extracted from the mammoth tusk, 40–50 polished ivory rods and 'two handsfull' of *Littorina* shells.

In preparation of a full entry on Paviland for the second part of the *Catalogue of Fossil Hominids* (Oakley, Campbell & Molleson 1971) relative dating tests were carried out on samples of material available for analysis.

Homo (Paviland 1), tibia: $F = 0.06\%$, $100F/P_2O_5 = 0.20$, e $U_3O_8 = $ nil, $N = 1.31\%$
Homo (Paviland 1), humerus: $F = 0.17\%$, $100F/P_2O_5 = 0.69$, $N = 2.65\%$ (w)
Ursus bone: $F = 0.048\%$, $100F/P_2O_5 = 0.15$, e $U_3O_8 = $ nil, $N = 0.93\%$ (w)
Mammuthus dentine (ivory rod): $F = 0.064\%$, $100F/P_2O_5 = 0.17$, $N = 0.13\%$ (w)
See Table 6, p. 30.

Without a longer series of samples for analysis it was not possible to infer that the human skeleton was appreciably later than the mammalian faunal material.

The cave and remaining contents of Goat's Hole were excavated by Sollas (1913), who found nearly 800 flint and chert artefacts, and remains of a characteristic Upper Pleistocene fauna, including *Rangifer*, which indicated cold dry climatic conditions. As there was no clear stratification, the time-span during which the cave was occupied by man can only be inferred from the typology of the stone tools. Mousterian, Lower, Middle and Upper Aurignacian, Proto-Solutrean and Creswellian tool-types were represented (Garrod 1926).

As there seemed to be no archaeological means of deciding to which of these human cultures the Paviland skeleton might belong, I requested that samples of the lower limb-bones should be drilled from the sides of their medullary cavities and submitted for radiocarbon dating at the Research Laboratory of the British Museum. The Curator of the Geological Collections at the University Museum, Oxford, (where the Paviland skeleton is preserved) agreed to the necessary procedures being carried out in the Subdepartment of Anthropology, BM(NH), and in turn the Research Laboratory of the British Museum accepted the sample for radiocarbon dating. The resulting date of $18\,460 \pm 340$ bp was obtained from residual collagen separated from bulked samples of the tibiae and left femur of the skeleton (BM-374; Barker, Burleigh & Meeks 1969 : 289, Oakley 1968). This dating pointed to the burial having taken place around the time of the Last Glacial maximum, when according to the geological evidence (Bowen 1970) glacier-ice was only about 6 km north of Paviland.

Molleson (1976) has argued that the date of the Paviland burial may not have been the date when the cave was actively occupied by the Palaeolithic hunters whose débris accumulated there. In an attempt to verify this she obtained a humerus of *Bos primigenius* from Goat's Hole, Paviland, in the Sollas collection in the National Museum of Wales, and submitted it to the Research Laboratory of the British Museum for radiocarbon dating. The result obtained using collagen separated from this bone was $27\,600 \pm 1\,300$ bp (BM-1367, Molleson & Burleigh 1978). This date compares closely with that of the earlier Upper Palaeolithic from Kent's Cavern (Davidson 1974) and can be seen as remarkably consistent with that part of the Paviland artefact collection which has been compared with the industry from Illsen Höhle near Leipzig, dated by palaeobotanical evidence to about 30 000 bp (McBurney 1965).

References: Barker, Burleigh & Meeks 1969 : 289, Bowen 1970, Buckland 1823 : 82–98, Davidson 1974, Garrod 1926 : 49–64, McBurney 1965, Molleson 1976 : 112–6, Molleson & Burleigh 1978, Oakley 1968, Sollas 1913.

PILTDOWN (Sussex). In 1950 it was shown by the fluorine method of relative dating that the Piltdown mandible and cranial bones were considerably younger geologically than the Lower and Middle Pleistocene fossils said to have been found at the same site. Assuming that they were genuine finds, the hominoid remains therefore could not be older than Upper Pleistocene, but it was noted that drill-holes into the teeth revealed that they were 'apparently no more altered than the dentine of recent teeth from the soil' (Oakley & Hoskins 1950 : 381). In 1953, J. S. Weiner, after reviewing this evidence in the light of anatomical considerations, suggested that the mandible

was that of a recent ape which had been broken and stained to resemble a fossil, and the teeth artificially abraded to suggest wear through the human type of mastication. According to this hypothesis, the fraudulent jaw-bone had been placed in the Piltdown gravel pit so as to appear associated with fragments of a thick human cranium of presumed great antiquity.

Determination of the organic content and redetermination of the fluorine content of these specimens, together with evidence obtained from a detailed anatomical analysis of the teeth, confirmed this hypothesis (Weiner, Oakley & Le Gros Clark 1953).

	Fluorine % (1950)	Fluorine % (1953)	Nitrogen %	Carbon %
Piltdown mandible	0.2 ± 0.2	< 0.03	3.9	14.5
Piltdown cranium	0.2 ± 0.1	0.10 ± 0.01	1.4	5.3
Modern bone		0.03 ± 0.01	4.0	14.0

The mandible had the composition of modern bone, whereas the cranial fragments appeared 'slightly fossilized'.

In 1953–55, the possibility of dating the Piltdown bones by the radiocarbon method was not seriously considered because it would have involved total destruction of the specimens to provide the minimum quantity of carbon (2 gm) then demanded by radiocarbon laboratories for a single determination. By 1959 improvement of technique made it possible to attempt ^{14}C dating on the basis of much smaller quantities. Powder samples of the Piltdown mandible and right parietal bone were then submitted to the late Professor H. de Vries of Groningen, after repeated washing in acetone and then in warm water (at 70°C), which was carried out under rigorous conditions in the Department of the Government Chemist, London. The nitrogen content of the samples proved to be the same before and after submission to this treatment, proving their freedom from nitrogenous contaminants, such as glue, gelatine and celluloid. Their nitrogen content represents the bone protein (collagen), which is not removable by acetone or warm water. This is the material which provided the carbon whose radioactivity was then measured in the Groningen Laboratory.

Professor de Vries reported that the sample of Piltdown mandible was burnt without further pre-treatment. The sample of the Piltdown cranium was dissolved in hydrochloric acid; the fraction which was not precipitated by alkali was dated. Both samples gave a very small amount of carbon dioxide, corresponding to about 0.1 gm of carbon. The radiocarbon dates obtained were as follows:

Piltdown mandible (GrN-2204) 500 ± 100 years bp (= AD 1450)
Piltdown skull (GrN-2203) 620 ± 100 years bp (= AD 1330)

These results show that both the mandible and the cranium are of Holocene age, conclusions not inconsistent with the relative dating published in 1955 when it was reported that 'the low fluorine content of the skull indicates that it is more probably Post-Pleistocene than Pleistocene in age' (Oakley 1955c : 257). The indication that the mandible was several centuries old raised the question of its origin. When Sir Wilfrid Le Gros Clark established that it was the jaw-bone of an orang-utan (and therefore came from Borneo or Sumatra), it was assumed to be a zoological collectors' piece. It seemed very unlikely that an orang-utan jaw-bone falling into the hands of a collector would prove to be several centuries old, but not impossible because the Dyaks of Borneo are known to keep orang-utan skulls as trophies in their long-houses for generations, although the chance of a dealer obtaining such a highly treasured object directly from the living Dyaks is negligible. However, Tom Harrison pointed out to me that a number of sub-recent bones of orang-utan, obtained in Sarawak in various circumstances, were brought back to Britain in 1875 by A. H. Everett. Some were obtained from caves whose contents were washed by Chinese gold-workers. A large part of Everett's collection (including one specimen No. 55 which appears to have been a trophy skull) was presented to the Department of Zoology of the BM(NH). All the orang-utan skulls and jaws listed as received by the Museum in 1879 can be accounted for, but Harrison believed that Everett brought back much more material to Britain, and what became of it is unknown.

Comparison of the Piltdown jaw-bone with one of the 'sub-recent' orang-utan jaw-bones in the Everett Collection (No. 100 in Everett's list, recorded as 'doubtfully from a cave in Sarawak', registered as ZD.84.10.30.2) has brought out a number of points of resemblance. Not only is the nitrogen content of the 'antique' Sarawak specimen (4·6%) undiminished in comparison with modern bone (4·0 to 5·3% in one series), but also the surface of the bone is in the same finely crackled condition. Attempts to reproduce the appearance of the Piltdown mandible by artificial treatment of modern bones failed in just this respect (Weiner et al. 1955 : pl. 27). The banded condition of the collagen fibrils in the Piltdown mandible, as revealed by the electron microscope (Weiner et al. 1955 : pl. 30, fig. 11), indicates that it was not boiled by the forger.

According to the radiocarbon dating of a sample of the right parietal bone, the Piltdown cranium appears to be only marginally older than the mandible. This is surprising in so far as in 1953 it was proved that all the cranial fragments had been artificially stained to match the gravel, so it became clear that like the mandible they were fraudulent introductions at the site.

Evidence was found by G. F. Claringbull & M. H. Hey (1955) that the Piltdown cranial bones had received very severe chemical treatment in an attempt to make them appear fossilized. The calcium phosphate in the bones had been partially altered to calcium sulphate (gypsum). Such alteration is unknown under natural conditions, but does occur when bones are artificially stained by an acid iron sulphate solution. Experiments showed that this alteration only occurs if the bone has the porosity due to partial loss of the organic matrix. Thus the Piltdown cranial bones are evidently of moderate antiquity. Presumably they were parts of a skull selected for the hoax on account of its exceptional thickness from amongst a series obtained in the excavation of some early burial ground. The fluorine content of the unaltered portion of the bone substance of the cranium also indicates moderate antiquity, but unless one knows the source of a bone precise relative dating by fluorine content is impossible. However, in many British and foreign cemeteries bones less than a thousand years old can be found with the same fluorine content (0·1%).

Although the difference in the radiocarbon ages of the cranial bone and the mandible is less than might have been expected in view of their contrasting states of preservation, it should be borne in mind that whereas a bone that has been buried in the ground for a few centuries may have become porous and 'sub-fossil' (with some absorbed fluorine), a bone of equal antiquity that has been preserved in air, for example on the floor of a dry cave, in a building or in a reliquary, may have retained the composition of recent bone.

References: Baynes-Cope 1955, Claringbull & Hey 1955, de Vries & Oakley 1959, Everett 1879 : 149, Oakley 1955*b, c*, Oakley & Hoskins 1950, Weiner, Oakley & Le Gros Clark 1953, Weiner *et al.* 1955.

STRUMPSHAW (Norfolk). A cranium regarded as representing one of the River Valley people of the Téviec Group was recorded from Strumpshaw by Wells (1961). Analysis suggests that the skull is of early Holocene age.

Strumpshaw skull: $F = 0·17\%$, $100F/P_2O_5 = 0·74$, $N = 2·94\%$ (w)
Skull of *Bos*, Early Iron Age, Stiffkey, Norfolk: $F = 0·03\%$, $100F/P_2O_5 = 0·1$, $N = 1·21\%$ (w)

References: Oakley 1971*a* : 39, Wells 1961.

SUN HOLE (Somerset). In 1926–28 excavations by the Bristol University Speleological Society in Sun Hole, a cave on the north side of Cheddar Gorge, Mendip Hills, produced a human radius bone (Sun Hole 2) at a depth of 5 ft (1·5 m) in solifluded scree overlying red cave-earth.

Sun Hole 2: $F = 1·09\%$, $100F/P_2O_5 = 4·86$, $N = 1·65\%$
Rangifer bone at same level: $F = 0·64\%$, $100F/P_2O_5 = 2·64$, $N = 3·16\%$
See Table 6, p. 30.

^{14}C dating of residual collagen in a bone of *Ursus arctos* from the B2 complex of solifluded screes in Sun Hole gave a date of $12\,378 \pm 150$ bp (BM-524).

References: Barker, Burleigh & Meeks 1971 : 168, Campbell 1977 : 51–55, Molleson 1977 : 89, 91, Oakley 1971*a* : 39.

Table 4 Analyses, British Isles, Lower Pleistocene. Piltdown I and II.

BM(NH) Lab. No.	Locus and Description	F%	P_2O_5%	$\dfrac{F\%}{P_2O_5\%} \times 100$	N%	$e\,U_3O_8$ ppm	U ppm	BM(NH) Register No.
'Piltdown I' collection, specimens inferred to be from Red Crag, Suffolk:								
P. 6	*Anancus* cf. *arvernensis* molar enamel	1·9	23·0	8·3	—	11	24	E.595
P. 25	*Anancus* cf. *arvernensis* molar enamel	2·3	36·0	6·4	nil	97	80	E.622
P. 26	*Dicerorhinus* cf. *etruscus* premolar enamel	2·0	24·0	8·3	nil	68	—	E.623
P. 48a	*Dicerorhinus* cf. *etruscus* molar dentine	2·07	30·3	6·8	—	40	—	E.624
P. 48b	*Dicerorhinus* cf. *etruscus* molar dentine	—	—	—	—	11	—	E.624
'Piltdown II' collection included one specimen inferred to be from Red Crag, Suffolk:								
P. 47a	*Dicerorhinus* cf. *etruscus* molar dentine	2·65	27·9	9·5	—	18	—	E.649
P. 47b	*Dicerorhinus* cf. *etruscus* molar enamel	—	—	—	—	<1	—	E.649

SWANSCOMBE (Kent). The Swanscombe skull, now referred to *Homo sapiens steinheimensis*, was discovered by A. T. Marston in 1935 and 1936, at a depth of 24 ft (7·3 m) below the surface in Barnfield gravel-pit, about $\frac{1}{2}$ mile (0·8 km) NW of Swanscombe Church. It lay near the base of the Upper Middle Gravels of the 100-ft terrace of the river Thames. The occipital bone was found on 29th June 1935 and the left parietal on 15th March 1936. The right parietal was found by A. Gibson on 30th July 1955.

The sandy gravel in which the skull bones were embedded contains a mammalian fauna including *Palaeoloxodon antiquus*, and has been accepted as belonging to the Hoxnian (= Holsteinian) interglacial.

Swanscombe, occipital: $F = 1·7\%$, $100F/P_2O_5 = 6·1$, $e\ U_3O_8 = 27$ ppm, $N = 0·18\%$

Swanscombe, l. parietal: $F = 1·4\%$, $100F/P_2O_5 = 5·9$; $F = 1·5\%$, $1·8\%$ (mean $1·6\%$); $e\ U_3O_8 = 11$ ppm.

Swanscombe, r. parietal: $F = 1·9\%$, $100F/P_2O_5 = 6·7$, $e\ U_3O_8 = 40$ ppm, $N = 0·09\%$

Associated mammalian bones: $F = 1·7–2·3\%$, $100F/P_2O_5 = 5·7–6·8$, $e\ U_3O_8 = 10–47$ ppm, $N = $ trace–$0·2\%$, $CaCO_3 = 6–9\%$

By measuring the thorium 230/uranium 234 and protactinium 231/uranium 235 activity ratios in a sample of bone collected from the Middle Gravels by Desmond Collins (sample 39), Barney J. Szabo (U.S. Geological Survey, Denver, Colorado) has obtained a provisional uranium-series dating of more than 272 000 years.

References: Molleson 1977 : 84–85, 88, Oakley 1971*a* : 40–41, Oakley & Gardiner 1964, 1968, Szabo & Collins 1975.

THATCHAM (Berkshire). Part of an adult human humerus was found on a Maglemosian (Meso-lithic) site at Thatcham near Newbury in 1959. It lay in reworked algal marl of Boreal age over-lying peat and containing wood which was dated by ^{14}C as 9490 ± 160 bp (Q-652).

Thatcham 1 bone: $F = 0·69\%$, $100F/P_2O_5 = 2·5$, $e\ U_3O_8 = 5$ ppm, $N = 1·74\%$

Cervid antler: $F = 0·7\%$, $100F/P_2O_5 = 2·4$, $e\ U_3O_8 = 7$ ppm, $N = 1·28\%$

Reference: Oakley 1971*a* : 41.

WALBROOK (City of London). An adult human frontal bone was found in a layer of gritty earth, 17 ft (5 m) below the surface, during excavation of shafts on the site of Western Union House, in the Walbrook Valley, London EC2 in 1944.

Walbrook 1 bone: $F = 0·63\%$, $100/P_2O_5 = 2·57$, $e\ U_3O_8 = $ nil, $N = 2·6\%$

F. E. Zeuner suggested that this bone was of Upper Pleistocene age, but an early Holocene date seems more probable in view of the moderately high nitrogen content and the lack of uranium.

Reference: Zeuner & Weiner 1947.

WESTLEY (Suffolk). The discovery at Westley near Bury St Edmunds in 1882 of a fragmentary human skull with sapient frontale, embedded in brickearth containing Acheulian hand-axes, was for long regarded as an indication that *Homo sapiens* had been in existence since Middle Pleistocene times. Application of the fluorine-dating method in 1950 left no doubt that the skull was part of an intrusive burial of Holocene age. The analyses were made by Dr C. R. Hoskins in the Depart-ment of the Government Chemist.

Westley skull: $F = 0·1–0·2\%$, $100F/P_2O_5 = 0·5–0·9$

Local Pleistocene bones: $F = 0·8–2·1\%$, $100F/P_2O_5 = 3·3–7·2$

Reference: Baden-Powell & Oakley 1953.

WHALEY (Derbyshire). Human skull fragments were reported from cave-earth in rock-shelter no. 2 at Whaley in 1947. According to A. L. Armstrong, the bones represented a ceremonial burial associated with Creswellian culture. J. B. Campbell reported (*in lit.*) that T. G. Manby was

Table 5 Analyses, British Isles, Middle Pleistocene. Swanscombe.

BM(NH) Lab. No.	Locus and Description	CFH No.	F%	P₂O₅%	$\frac{F\%}{P_2O_5\%} \times 100$	CaCO₃%	N%	e U₃O₈ ppm	Collection & BM(NH) Register No.
Lower Gravel, Swanscombe:									
S. 1	*Dama* humerus	—	2·0	32·0	6·3	17	—	15	M.16500
S. 2	*Dama* incisor (osteodentine)	—	2·8	35	8·0	6	—	—	M.16499
S. 36	*Dama* vertebra	—	2·1	34·0	6·2	10	—	14	M.16511
S. 41a	*Dama* antler, compact zone	—	2·2	29·0	7·6 }	11	—	—	} A. T. Marston Coll., BM(NH) unreg.
S. 41d	*Dama* antler, cancellar core	—	2·6	30·0	8·7 }		—	—	
S. 82	*Dama* antler	—	—	—	—	—	—	9 }	} J. Wymer Coll., BM(NH) unreg.
S. 83	*Dama* antler	—	—	—	—	—	—	14 }	
S. 48	fragment of ungulate limb-bone	—	2·4	33·2	7·4	—	0.20	42	E.2707
Lower Loam, Swanscombe:									
S. 16	*Panthera* humerus	—	1·7	32·5	5·2	9	—	15	M.16501
Middle Gravels, Swanscombe:									
S. 3	*Dama* metapodial	—	2·3	34	6·8	6	—	—	M.16510
S. 37	*Bos* rib	—	2·0	30	6·7	9·4	trace	32	E.2710
S. 18	*Bos* limb-bone	—	2·0	32	6·3	—	—	21 }	} K. P. Oakley Coll., BM(NH) unreg.
S. 19	rolled bone fragment	—	1·7	30	5·7	8	—	— }	
S. 4	*Homo* occipital	Swl	1·7	27·8	6·1	9·0	0·18 (w)	27	} A. T. Marston Coll., M.15709
S. 17	*Homo* left parietal	Swl	1·5	24	5·9	—	nil (w)	11 }	
S. 17	*Homo* left parietal	Swl	1·4	—	—	—	—	—	
S. 53	*Homo* right parietal	Swl	1·9	28·2	6·7	6·9	0·09 (w)	40	J. Wymer & A. Gibson Coll., EM.40
S. 78	nine mammal bone fragments from Middle Gravels, Swanscombe	—	—	—	—	—	0·20	47	BC/23
S. 79		—	—	—	—	—	—	21	BC/23
S. 80		—	—	—	—	—	—	17	AB/1–2
S. 81		—	—	—	—	—	—	27	CD/2–3
S. 84		—	—	—	—	—	—	29	AB/2–3
S. 85		—	—	—	—	—	—	31	BC/2–3
S. 86		—	—	—	—	—	—	22	—
S. 87		—	—	—	—	—	—	32	D1/3–4
S. 88		—	—	—	—	—	—	35	—

(S. 78–S. 88 collection entries braced: from J. Wymer's excavations, 1955)

Table 6   Analyses, British Isles, Late Upper Pleistocene and Early Holocene.

BM(NH) Lab. No.	Locus and Description	CFH No.	F%	P_2O_5%	$\frac{F\%}{P_2O_5\%} \times 100$	$CaCO_3$%	N%	$e\ U_3O_8$ ppm	^{14}C yrs bp	Collection & Register No.
C. 21	Aveline's Hole, *Homo* cranium	AH1	0·47	23·7	1·98	13·0	3·8 (w)	2	—	W. Buckland Coll., BM(NH) E.11.6.257
C. 19	Aveline's Hole, *Homo* mandible	AH2	0·29	27·8	1·04	20·5	1·8 (w)	nil	—	R. Bright Coll., BM(NH) E.504
C. 24	Aveline's Hole, *Homo* cranium	AH9	0·48	21·0	2·28	6·3	3·47	—	—	Wells Museum, 174
C. 25	Aveline's Hole, *Homo* metacarpal	—	0·2	21·6	0·92	8·3	3·49	nil	—	Univ. Bristol Spel. Soc., 133
—	Aveline's Hole, *Homo* femur	—	—	—	—	—	—	—	9114±110 (BM-471)	Univ. Bristol Spel. Soc., unreg.
C. 26	Aveline's Hole, mammal bone	—	0·07	27·1	0·26	6·0	2·9	nil	—	Univ. Bristol Spel. Soc., 139
C. 27	Aveline's Hole, mammal bone	—	—	—	—	—	3·0 (w)	—	—	Univ. Bristol Spel. Soc., unreg.
C. 30	Badger Hole, *Crocuta* bone	BH3	—	—	—	—	2·5	—	—	K. P. Oakley Coll., BM(NH) unreg.
C. 32	Badger Hole, *Homo* cranium	BH1	nil	22·2	0	8·1	0·93	nil	—	Wells Museum, 228
C. 48	Badger Hole, *Homo* mandible	—	—	—	—	—	1·58	—	—	Univ. Bristol Spel. Soc., 226
S. 39	Baker's Hole, *Mammuthus* limb-bone	—	1·2	28	4·3	12	0·32 (w)	15	—	F. N. Haward Coll., BM(NH) unreg.
C. 15	Flint Jack's Cave, *Homo* cranium	FJC1	0·05	30·6	0·17	8·7	1·36 (w)	—	—	R. Pavey Coll., BM(NH) M.16796
C. 16	Flint Jack's Cave, *Homo* cranium	FJC2	0·12	27·8	0·44	12·9	1·25 (w)	—	—	R. Pavey Coll., BM(NH) M.16795

Table 6, cont.

BM(NH) Lab. No.	Locus and Description	CFH No.	F%	P₂O₅%	$\frac{F\%}{P_2O_5\%}\times100$	CaCO₃%	N%	e U₃O₈ ppm	¹⁴C yrs bp	Collection and Register No.
C. 3	Gough's Cave, *Homo* right talus	GC1	0·25	10·5	2·4	55	1·83 (w)	nil	—	R. C. Gough Coll., Gough's Cave Museum, 1.1/29
C. 2	Gough's Cave, *Homo* molar (M₁) dentine	GC1	—	—	—	—	2·7	—	—	R. C. Gough Coll., Gough's Cave Museum, 1.1/5 or 1.1/6
C. 78	Gough's Cave, *Homo* left tibia	GC1	—	—	—	—	1·74–3·95 (21 samples)	—	9080±150 (BM-525)	R. C. Gough Coll., Gough's Cave Museum, 1.1/35
C. 79	Gough's Cave, *Homo* right tibia	GC1	—	—	—	—	0·8, 1·5	—	—	R. C. Gough Coll., Gough's Cave Museum, 1.1/27
C. 80	Gough's Cave, *Homo* left femur	GC1	—	—	—	—	1·5, 3·9	—	—	R. C. Gough Coll., Gough's Cave Museum, 1.1/34
C. 4	Gough's Cave, *Homo* cranium	GC3	—	—	—	10·7	2·9	—	—	R. F. Parry Coll., Gough's Cave Museum, 1.1/1
C. 33	Gough's Cave, *Homo* mandible	GC6	0·47	25·78	2·16	14·07	3·02	6	—	R. F. Parry Coll., Gough's Cave Museum, 1.1/3
C. 34	Gough's Cave, *Homo* parietal	GC7	0·09	6·57	1·37	63·42	0·71	nil	—	Gough's Cave Museum, 1.1/2
C. 6	Gough's Cave, level 11, *Ursus* radius	—	0·24	27·0	0·89	10·32	3·0	nil	—	Gough's Cave Museum, 1.2/63a
C. 7	Gough's Cave, level 12, *Ursus* radius	—	—	—	—	—	2·7	—	—	Gough's Cave Museum, 1.2/63
C. 8	Gough's Cave, above⁶ GC1, *Rangifer* mandible	—	0·14	16·3	0·9	28·2	1·5	nil	—	
C. 9	Gough's Cave, above⁶ GC1, *Rangifer* molar dentine	—	—	—	—	26·3	1·6	—	—	Gough's Cave Museum, 1.2/25
C. 10	Gough's Cave, level 18, *Equus* talus	—	0·29	20·44	1·42	21·4	2·1	4	—	Gough's Cave Museum, 1.2/2
M. 16	Kent's Cavern, *Homo* humerus	KC2	0·1	37	0·3	—	1·38	nil	—	W. Pengelly Coll., BM(NH) M.576
M. 40	Kent's Cavern, *Homo* ulna	—	0·01	27·9	0·03	5·55	2·11	nil	—	W. Pengelly Coll., BM(NH) 16769
M. 46	Kent's Cavern, Proto-Solutrean, *Ursus* bone	—	—	—	—	—	3·53	—	—	J. B. Campbell Coll. 1562
AY	Langwith Cave, *Homo* cranium	L1	—	—	—	—	2·7	—	—	Oxford Coll. E.11.6/258, BM(NH)
M. 21	Mother Grundy's Parlour, *Homo* molar dentine	MGP1	—	—	—	—	0·95 (w)	—	—	W. B. Dawkins Coll., Manchester Museum P4410
M. 22	Probably Mother Grundy's Parlour, *Meles* molar dentine	—	—	—	—	—	1·20 (w)	—	—	W. B. Dawkins Coll., Manchester Museum P2961

⁶According to a register of the Gough's Cave finds shown to the author in 1954 by the late Curator, Mr Gerald Robertson, the reindeer bone 1.2/25 had been found 'above' the skeleton of Cheddar Man. As it is now generally agreed that this skeleton (GC1) represents an early Post-Pleistocene burial, any overlying bones could easily have been derived from a pre-existing Pleistocene deposit through which the burial was made (Prof. D. T. Donovan *in lit.* 26 June 1975).

Table 6, cont.

BM(NH) Lab. No.	Locus and Description	CFH No.	F%	P_2O_5%	$\frac{F\%}{P_2O_5\%} \times 100$	$CaCO_3$%	N%	$e\,U_3O_8$ ppm	^{14}C yrs bp	Collection and Register No.
E0	Paviland, *Homo* humerus	P1	0·17	25·1	0·69	—	2·65 (w)	nil	—	W. Buckland Coll., Oxford Univ. Museum Q1/1–1/28
K1	Paviland, *Homo* tibia	P1	0·06	29·8	0·20	7·25	1·31	nil	18 460 ± 340 (BM-374)	
PJ	Paviland, *Bos primigenius* humerus	—	—	—	—	—	1·07 (w)	—	27 600 ± 1300 (BM-1367)	W. J. Sollas Coll., Nat. Mus. Wales
EP	Paviland, *Ursus* metapodial	—	0·048	32·3	0·15	—	0·93 (w)	nil	—	F. Corner Coll. 291, BM(NH) unreg.
FA	Paviland, *Mammuthus* dentine	—	0·064	36·8	0·17	—	0·13 (w)	—	—	P. Egerton Coll., BM(NH) M.416
C. 17	Pin Hole Cave, *Homo* ilium	PHC1	0·06	21·03	0·27	16·24	3·41	nil	—	A. L. Armstrong Coll, BM(NH) EM 607
C. 18	Pin Hole Cave, carnivore phalange	—	0·54	21·5	2·51	8·39	1·94	nil	—	A. L. Armstrong Coll., BM(NH) M. 36779
PI	Robin Hood's Cave, *Homo* mandible c. 1 m below travertine floor	—	—	—	—	—	1·11	—	—	Creswell Crags Visitor Centre Museum (1974 coll.)
—	Robin Hood's Cave, *Equus przewalskii* metacarpal, layer OB	—	—	—	—	—	—	—	10 390 ± 90 (BM-603)	J. B. Campbell Coll. (1969), Creswell Crags Visitor Centre Museum
M. 41	Robin Hood's Cave, *Homo* frontale from layer OB	RHC1	—	—	—	—	3·89	—	—	J. B. Campbell Coll. (1969) No. 465, Creswell Crags Visitor Centre Mus.
M. 43	Robin Hood's Cave, *Homo* maxilla from layer E (tip)	—	—	—	—	—	3·81	—	—	J. B. Campbell Coll. (1969) No. 466, Creswell Crags Visitor Centre Mus.
M. 54	Robin Hood's Cave, *Homo* cervical vertebra from layer E (tip)	—	—	—	—	—	4·12	—	—	J. B. Campbell Coll. 1969, Creswell Crags Visitor Centre Mus.
M. 55	Robin Hood's Cave, *Homo* calotte from layer E (tip)	—	—	—	—	—	3·94	—	—	
C. 39	Sun Hole, 5th ft *Homo* radius	SH2	1·09	22·44	4·86	25·02	1·65	5	—	Univ. Bristol Spel. Soc. M.5.13/24
—	Sun Hole, 5th ft (B2-7 in up. thermoclastic scree), *Ursus arctos* humerus	—	—	—	—	—	—	—	12 378 ± 150 (BM-524)	J. B. Campbell Coll.
C. 40	Sun Hole, 1st ft mammal bone	—	0·65	17·39	3·74	44·08	2·71	nil	—	Univ. Bristol Spel. Soc. unreg.
C. 42	Sun Hole, 3rd ft *Rangifer* talus	—	—	—	—	—	2·0	nil	—	Univ. Bristol Spel. Soc. M.5.2/12
C. 43	Sun Hole, 4th ft mammal bone	—	—	—	—	—	2·5	nil	—	Univ. Bristol Spel. Soc. unreg.
C. 44	Sun Hole, 5th ft *Rangifer* femur	—	0·64	24·25	2·64	14·50	3·16	nil	—	Univ. Bristol Spel. Soc. M.5.2/2
C. 45	Sun Hole, 6th ft mammal bone	—	—	—	—	—	1·3	nil	—	Univ. Bristol Spel. Soc. unreg.
C. 46	Sun Hole, 7th ft *Equus* humerus	—	—	—	—	—	3·2	nil	—	Univ. Bristol Spel. Soc. M.5.2/27

'unable to recognize any Creswellian material amongst the excavated industry from this site'. Analyses indicate that the skeletal material is not older than Early Holocene.

Whaley 1: $F = 0.12\%$, $100F/P_2O_5 = 0.5$, $N = 2.29\%$ (w)

References: Oakley 1971*a* : 42–43, Radley 1967.

France

AURIGNAC (Haute-Garonne). Human mandible of Aurignacian or Neolithic age from grotte d'Aurignac collected by Rev. W. S. King before 1871 (reg. no. EM 326): $F = 0.035\%$, $100F/P_2O_5 = 0.12$, $N = 1.33\%$.

In this cave 17 human skeletons representing a collective burial dating from the Neolithic period occurred on top of a cave deposit of Upper Palaeolithic age. Some detached human bones were found mixed with bones of Upper Pleistocene mammals in the substratum of the burial chamber. Chemical analyses in the last century proved that the Neolithic human bones contained no more gelatine (cf. nitrogen) than the Pleistocene mammal bones. See Table 9, p. 36.

References: Lartet 1861 : pl. 10, fig. 1; Oakley 1964*c* : 106–107.

BRUNIQUEL (Tarn et Garonne). Humerus of *Homo sapiens sapiens* from Upper Magdalenian layer in Trou des Forges, Bruniquel (reg. no. EM 546): $F = 0.44\%$, $100F/P_2O_5 = 1.41$, e $U_3O_8 = $ nil, $N = 1.64\%$. See Table 9, p. 36.

The ^{14}C age of the collagen fraction of associated biserial antler fragments is 11 750 ± 300 bp (BM-302).

References: Barker, Burleigh & Meeks 1969 : 283, Petit-Maire *et al.* 1971 : 88–91

LA CHAPELLE-AUX-SAINTS (Corrèze). Postcranial bone of the neandertal skeleton La Chapelle-aux-Saints 1: $F = 0.358\%$, $100F/P_2O_5 = 2.2$, e $U_3O_8 = 12$ ppm, $N = 2.12\%$ (w). See Table 9, p. 36.

The relatively high nitrogen content (representing residual collagen) is probably to be explained by the fact that the burial was covered by stony clay. Water rising through the rock floor of the grave evidently carried elements of the uranium series.

Reference: Petit-Maire *et al.* 1971 : 98–99.

CHÂTELPERRON (Allier). A human calvaria from Châtelperron is preserved in the Wellcome Historical Medical Museum, London. It was widely supposed that this had been obtained from an Aurignacian horizon in the Grotte des Fées in the Châtelperron province, 5 km east of Jaligny (Allier), which was excavated by Bailleau (1868–70). As indicated by Vallois & Movius (1953 : 105), and confirmed personally by H. Delporte (*fide* 1963), the calvaria in the Wellcome Museum did not come from the Grotte des Fées but from an open site in the same region. Although its precise antiquity is doubtful, the following comparative analyses of bones from Châtelperron and neighbourhood suggest that it is probably of Late Pleistocene age.

Châtelperron *Homo* calvaria: $F = 0.15\%$, $100F/P_2O_5 = 0.5$, e $U_3O_8 = 13$ ppm, $N = 1.64\%$ (w)
Neolithic *Homo* cranium: $F = 0.12\%$, $100F/P_2O_5 = 0.5$, e $U_3O_8 = 8$ ppm, $N = 3.24\%$
Perigordian *Ursus* bone: $F = 0.07\%$, $100F/P_2O_5 = 0.4$, e $U_3O_8 = 7$ ppm, $N = 1.49\%$

References: Petit-Maire *et al.* 1971 : 100, Vallois & Movius 1953 : 105.

CRO-MAGNON (Dordogne). Postcranial bone of *Homo sapiens sapiens* skeleton, 'Le Vieillard' (= Cro-Magnon 1): $F = 0.12\%$, $100F/P_2O_5 = 0.38$, e $U_3O_8 < 1$ ppm, $N = 0.455\%$ (w). See Table 9, p. 36.

The low fluorine content is typical of bones in limestone cave deposits of Late Pleistocene age. The nitrogen content (representing residual collagen) is unusually low, probably because the containing layer was in contact with gravel and well drained.

Reference: Petit-Maire *et al.* 1971 : 104–105.

LA DENISE (Haute-Loire). In 1843 or 1844 an adult hominid frontal bone was found by Adsclénard in deposits overlying the southern slopes of an ancient volcano, La Denise, 5 km from Le Puy-en-Velay, Haute-Loire. Later, other hominid bones were found by Aymard, Pichot and Gervais. Although no fossil mammalia were found in association with the hominid bones, deposits (breccias) on the eastern slopes of the volcano contain *Equus stenonis*, *Dicerorhinus etruscus* and *Archidiskodon meridionalis*, indicating their Lower Pleistocene antiquity.

The age and authenticity of the hominid remains have for long been controversial. Application of analytical methods of relative dating showed that none is contemporaneous with the Lower Pleistocene fauna.

Homo, frontal (first discovery): $F = 0.06\%$, $100F/P_2O_5 = 0.19$, $N = 1.68\%$ (w), $e\ U_3O_8 =$ nil
Homo, ilium, Aymard block: $F = 0.06\%$, $100F/P_2O_5 = 0.25$, $N = 4.35\%$, $e\ U_3O_8 =$ nil
Homo, ilium, Pichot block: $F = 0.07\%$, $100F/P_2O_5 = 0.30$, $N = 4.21\%$, $e\ U_3O_8 =$ nil
Dicerorhinus, occipital, breccias: $F = 1.53\%$, $100F/P_2O_5 = 4.89$, $N = 0.74\%$, $e\ U_3O_8 =$ nil
See Table 7, p. 34.

The high nitrogen content shown by the bones from the Aymard and Pichot blocks is comparable with that of recent (Holocene) bones. Brown earth with which the bones were covered suggests that they represent burials (M. R. Gounot, *in lit.*). The lower nitrogen content of the frontal indicates that it may be slightly more ancient than the other bones, but probably they are all of Holocene age.

References: Heintz & Oakley 1969 : 2873–2874, Petit-Maire *et al.* 1971 : 107.

LA FERRASSIE (Dordogne). Cancellous bone from foot of neandertal skeleton, La Ferrassie 1: $F = 0.06\%$, $100F/P_2O_5 = 0.37$, $e\ U_3O_8 =$ nil, $N = 1.26\%$ (before washing), $N = 2.74\%$ (after washing). See Table 9, p. 36.

The composition of this bone is typical of skeletal material from French limestone cave deposits of Upper Pleistocene age. The nitrogen content appears to form a higher proportion of the sample *after* washing, indicating that much non-nitrogenous material was removed by the process of elutriation.

Reference: Petit-Maire *et al.* 1971 : 111–112.

FONTÉCHEVADE (Charente). In 1949 at the request of Mlle G. Henri-Martin the fluorine-dating method was applied to the Fontéchevade skulls. No one would question the provenance of Fontéchevade I and II found in 1947, now known as Fontéchevade 4 and 5 (Petit-Maire *et al.* 1971 : 115). They were unearthed from an Eemian (clayey gravel) deposit containing a Tayacian industry and sealed by a stalagmitic layer ('*plancher*') which in turn was overlain by layers containing Mousterian and Aurignacian industries. The analytical results obtained were instructive and worth recording here.

Fontéchevade 1 skull fragment, *Homo sapiens sapiens* (found before 1913), Aurignacian: $F = 0.05\%$, $100F/P_2O_5 = 0.2$, $N = 0.91\%$
Fontéchevade 4 (formerly I) calotte, *Homo* sp., Tayacian: $F = 0.4\%$, $100F/P_2O_5$ not determined.
Fontéchevade 5 (formerly II) calotte, *Homo* sp., Tayacian: $F = 0.5\%$, $100F/P_2O_5 = 2.4$, $N = 0.63\%$ (w), $U_3O_8 = 3.2$ ppm (determined as uranium $= 3.8 \pm 0.4$ ppm)
Mammal bones from Tayacian deposits, Niveau 2 (lower level): $F = 0.9\%$, $100F/P_2O_5 = 2.8$; $F = 0.7\%$, $100F/P_2O_5 = 2.3\%$, $N = 2.7\%$ (w), $e\ U_3O_8 = 6$ ppm.
Ditto, Niveau 1 (upper level): $F = 0.6\%$, $100F/P_2O_5 = 1.9$, $N = 1.87\%$, $e\ U_3O_8 =$ nil; $F = 0.5\%$, $100F/P_2O_5 = 1.7$, $N = 1.62\%$ (w)
Mammal bone from Mousterian sandy lens within stalagmite, interior of bone: $F = 0.05\%$, $100F/P_2O_5 = 0.2$; exterior of bone: $F = 0.2\%$, $100F/P_2O_5 = 0.7$
Mammal bone from Aurignacian breccia: $F = 0.1\%$, $100F/P_2O_5 = 0.6$
See Tables 8, p. 35, and 9, p. 36.

In this very small series of analysed bones there is only a slight increment in the fluorine content from Aurignacian and Mousterian deposits above, to the Tayacian layers below. The fluorine contents of Fontéchevade 4 and 5 are in conformity with the Tayacian controls. The

nitrogen contents of Tayacian and Aurignacian (Fontéchevade 1) bones are not significantly different. It is notable that the average fluorine content of the Mousterian bone places it between the Tayacian and the Aurignacian. The difference in fluorine content between the outer layer of this bone (0.2%F) and the interior (0.05%F) illustrates the importance of only using for comparison samples which are representative of the whole bone. Each of the samples of the Tayacian calottes submitted for analysis included the outer table and the diploic layer and was therefore fully representative.

A sample of charred bone from the Tayacian Niveau 2 (lower level) was found to contain an appreciably lower fluorine content (0.3%) than unburnt bones from the Tayacian layers (0.4–0.7%F). This is in accordance with expectations, for it has been found experimentally that charring inhibits subsequent fluorination of bone.

References: Henri-Martin 1951, Oakley & Hoskins 1951, Petit-Maire *et al.* 1971 : 113–115.

MOULIN-QUIGNON (Somme). In March 1863, the left side of a human mandible, with the second molar in place, was allegedly found by workmen at a depth of 4·5 m in high terrace gravels, containing extinct mammalia and palaeolithic hand-axes, exposed in the Moulin-Quignon pit near Abbeville (Somme). In the following year, a second mandible was reported from the same gravels. These discoveries led to heated controversy, but eventually the conclusion was reached that the Moulin-Quignon mandibles were not older than Neolithic and had been 'planted' by the gravel diggers wishing to please M. Boucher de Perthes. That the Moulin-Quignon mandibles were intrusive in the gravels has been confirmed by chemical assays of samples of these specimens taken by permission of the Director of the Musée de l'Homme, Paris, in 1950.

Moulin-Quignon mandible no. 1: $F = 0.12\%$, $100F/P_2O_5 = 0.4$, $N = 2.05\%$ (w)
Moulin-Quignon mandible no. 2: $F = 0.05\%$, $100F/P_2O_5 = 0.2$
Palaeoloxodon molar (dentine), Moulin-Quignon pit: $F = 1.7\%$, $100F/P_2O_5 = 5.1$
Homo sapiens, calvaria, Neolithic, Champs-de-Mars (Somme): $F = 0.05\%$, $100F/P_2O_5 = 0.2$
See Table 7, p. 34.

References: Delesse 1863, Oakley 1964c : 111–115, 117.

SOLUTRÉ (Saône). Although skeletal remains of more than 60 individuals of *Homo sapiens* were obtained from the early excavations at the open site, Crôt du Charnier, Solutré, the archaeological provenance of the material is uncertain; some of it may be Upper Palaeolithic, but most of it is now considered to be Neolithic or later.

Human mandible from early excavations: $F = 0.1\%$, $100F/P_2O_5 = 0.4$, e $U_3O_8 = $ nil, $N = 2.16\%$ (w)

A fragment of human mandible from the 1923 excavations was submitted by Dr Nicole Petit-Maire for determination of its nitrogen content, which proved to be 2.27%, high enough on an open site to allow reference to the Holocene.

Reference: Petit-Maire *et al.* 1971 : 177–178.

VEYRIER (Haute-Savoie). A skeleton and also cranial and post-cranial bones of 9 other individuals (*Homo sapiens*) were found in the Veyrier rock-shelters at Étrembières, Annemasse, Saint-Julien, during explorations in 1833–34, 1916 and 1934 onwards. With the exception of one humerus regarded as Holocene, this skeletal material has been attributed to the Magdalenian. Analyses indicated a wide range of collagen content in these bones.

Veyrier 1 (adult skeleton): $N = 3.73\%$ (w)
Veyrier human bones (14): $N = 1.2$–4.6%
Rangifer bone: $N = 2.61\%$ (w)
Rupicapra bone: $N = 2.81\%$

The fact that in no less than seven of the tested bones attributed to Magdalenian man, the nitrogen content proved to be 4% (a level common in modern bone) suggests that collagen may

Table 7 Analyses, France, Middle Pleistocene.

BM(NH) Lab. No.	Locus and Description	F%	P_2O_5%	$\frac{F\%}{P_2O_5\%} \times 100$	$CaCO_3$%	N%	$e\,U_3O_8$ ppm	Collection
F. 1	30 m or 45 m terrace of Somme, Abbeville, bovid bone	0·9	24	3·7	—	—	—	Musée de l'Homme
F. 2	30 m or 45 m terrace of Somme, Abbeville, 'Elephas' dentine	1·6	36	4·4	—	—	—	Musée de l'Homme
F. 3	Moulin-Quignon Palaeoloxodon dentine	1·7	33	5·1	—	—	—	Boucher de Perthes Coll., Musée de l'Homme
F. 52	Fissure deposit, E slopes, La Denise, Dicerorhinus occipital	1·53	31·29	4·89	9·8	0·74	nil	Musée Crozatier, Le-Puy-en-Velay, (Haute-Loire)
F. 72-F. 80	Sand-dune deposits, Mindelian? Terra Amata, nine mammal bones	—	—	—	—	nil	—	H. de Lumley Coll., Marseilles

Table 8 Analyses, France, early Upper Pleistocene.

BM(NH) Lab. No.	Locus and Description	CFH No.	F%	P$_2$O$_5$%	$\frac{F\%}{P_2O_5\%} \times 100$	CaCO$_3$%	N%	e U$_3$O$_8$ ppm	U ppm	Collection
F. 4	10 m terrace Somme, Menchecourt, *Equus* metatarsal	—	1·1	33	3·2	—	—	—	—	Musée de l'Homme
F. 5	10 m terrace Somme, Menchecourt, *Dicerorhinus* talus	—	1·2	35	3·4	—	—	—	—	Musée de l'Homme
F. 19	Tayacian cave-deposits, Fontéchevade, Bed E1′ *Homo* parietal	F5	0·5	21	2·4	—	0·63 (w)	3·2	3·8	Villebois-Lavalette, Charente. G. Henri-Martin Coll.
F. 23	Tayacian cave-deposits, Fontéchevade, Bed E0 *Homo* frontal	F4	0·4	—	—	—	—	—	—	Villebois-Lavalette, Charente. G. Henri-Martin Coll.
F. 18	Tayacian cave-deposits, Fontéchevade, Bed E1′ animal bone	—	0·5	29	1·7	9	1·62 (w)	—	—	Villebois-Lavalette, Charente. G. Henri-Martin Coll.
F. 17	Tayacian cave-deposits, Fontéchevade, Bed E1′ animal bone	—	0·6	31	1·9	—	1·87	nil	—	Villebois-Lavalette, Charente. G. Henri-Martin Coll.
F. 16	Clacto-Tayacian cave-deposits, Fontéchevade, Bed E2 animal bone	—	0·9	32	2·8	—	—	—	—	Villebois-Lavalette, Charente. G. Henri-Martin Coll.
F. 15	Clacto-Tayacian cave-deposits, Fontéchevade, Bed E2 *Dama* bone	—	0·7	30	2·3	—	2·72 (w)	6	—	Villebois-Lavalette, Charente. G. Henri-Martin Coll.
F. 25	Clacto-Tayacian cave-deposits, Fontéchevade, Bed E2 burnt animal bone	—	0·3	31·0	1·0	—	—	—	—	Villebois-Lavalette, Charente. G. Henri-Martin Coll.
F. 45	Clacto-Tayacian cave-deposits, Fontéchevade, Bed E2 animal bone	—	—	—	—	—	0·32	—	—	Villebois-Lavalette, Charente. G. Henri-Martin Coll.

Table 9   Analyses, France, late Upper Pleistocene

BM(NH) Lab. No.	Locus and Description	CFH No.	F%	P_2O_5%	$\dfrac{F\%}{P_2O_5\%} \times 100$	$CaCO_3$%	N%	$e\,U_3O_8$ ppm	^{14}C yrs bp	Collection and Register No.
F. 21	Aurignacian, Fontéchevade, *Homo sapiens sapiens* parietal	1	0·05	33·0	0·2	—	0·91 (w)	—	—	Dugonthier Coll.
F. 24	Aurignacian breccia, Fontéchevade, *Saiga* bone	—	0·1	16·5	0·6	—	—	—	—	G. Henri-Martin Coll.
F. 26	Mousterian stalagmite, Fontéchevade, *Equus* phalange, inner zone	—	0·05	29·0	0·2	—	—	—	—	G. Henri-Martin Coll.
	Ditto, outer zone	—	0·2	27·5	0·7	—	—	—	—	
F. 48	Magdalenian, Trou des Forges, Bruniquel, *Homo* humerus	—	0·44	31·1	1·41	10·89	1·64	nil	—	BM(NH) EM 546
—	Upper Magdalenian antler harpoon fragments, Trou des Forges, Bruniquel	Cf. B4-23	—	—	—	—	—	—	11 750±300 (BM-302)	F. de Lastic Coll., Musée de l'Homme
F. 34	Aurignacian, Grotte d'Aurignac, *Rangifer* antler	—	0·70	30·5	2·3	—	1·03	—	—	E. Lartet Coll., BM(NH)
F. 36	La Chapelle-aux-Saints, *Homo neanderthalensis* skeleton	LC-S1	0·36	16·5	2·2	7·0	2·12 (w)	12	—	Musée de l'Homme
F. 37	Cro-Magnon, evolved Aurignacian *Homo* 'Le Vieillard' skeleton	C-M1	0·12	31·4	0·38	3·05	0·49	<1	—	Musée de l'Homme
F. 38	La Ferrassie *Homo neanderthalensis* footbone	F1	0·06	16·3	0·37	5·9	2·74 (w)	nil	—	Musée de l'Homme

have survived longer in bones preserved under cold damp conditions. An additional factor may have been the sealing in of the bone pores by films of calcite.

Reference: Sauter 1971*b* : 184–186.

Germany

KÖSTRITZ (Thuringia). It is recorded that in 1820, in Winter's Gypsum Quarry at Köstritz, 30 km east of Jena, a Dr Schammerring collected a hominid parietal bone and femur fragment from a depth of about 15 m in diluvial deposits below bones of '*Rhinoceros*'.

Analyses did not suggest high antiquity: $F = 0.11\%$, $100F/P_2O_5 = 0.35$, e $U_3O_8 = 2$ ppm, $N = 1.6\%$ (w).

Radiocarbon dating of the residual collagen in the femur indicate that the bones are Post-Pleistocene: 1480 ± 125 bp (BM-373). The specimens are of historic interest in view of the early date at which E. F. von Schlotheim reported this alleged association of man with a locally extinct mammal. The specimens are preserved in the BM(NH), M 16805–6.

References: Barker, Burleigh & Meeks 1971 : 167, Gieseler 1971 : 196–197.

MAUER (Heidelberg). The Heidelberg mandible was found by a workman, D. Hartmann, and recognized by Professor O. Schoetensack on 21 October 1907. The discovery was made in Sandgrube Rösch, 900 m north of Mauer, 16 km SE of Heidelberg. Although the depth from the surface at which the jaw occurred was not recorded, Schoetensack (1908) published photographs on which its original position in the section is clearly indicated. The stratigraphical age of the Mauer Sands from which this historic specimen undoubtedly came is generally given as Cromerian, but in the opinion of Dr Karl D. Adam it is more precisely Mosbachian. Schoetensack recorded that a specimen of *Palaeoloxodon antiquus* occurred at the same level 11·5 m south from the human mandible.

In September 1965 I visited the Geologisch-Paläontologisches Institut of Heidelberg University, where the Mauer mandible is preserved, and through the courtesy of the Director, Professor Schonenberg, obtained samples of the mandible and of associated fauna for analysis in London. A microsample of the mandible was taken close to the symphysis and another microsample of the left first premolar.

Analyses for fluorine, phosphate and nitrogen of these samples were made in the Laboratory of the Government Chemist, London. Radiometric assays of the larger samples were carried out in the Subdepartment of Anthropology, BM(NH). Residual grains of the human mandible sample and of a small piece of associated bone of *Dicerorhinus etruscus* were sent to Dr R. L. Fleischer at the General Electric Research Laboratory at Schenectady, New York, who kindly determined their uranium content, here recalculated as U_3O_8.

	F%	100F/P₂O₅	N% (w)	U₃O₈ ppm
Homo heidelbergensis, mandible bone	1·13	10·0	0·08	6
Palaeoloxodon antiquus, Homo layer, bone	2·1	6·6	0·12	11[7]
Dicerorhinus etruscus, bone	2·2	7·4	<0·01	15

See Table 10, p. 38.

It is notable that the percentage of fluorine in the sample of the Mauer mandible that was analysed is lower than that in the sample of associated *Palaeoloxodon* bone, but the level of fluorination in the mandible, measured as $100F/P_2O_5$, is actually higher.

References: Gieseler 1971 : 197–198, Oakley 1958*a*, Schoetensack 1908.

OFNET (Bavaria). In 1908 R. R. Schmidt excavated a nest of about 30 human crania in the cave of Grosse Ofnet, 4 km SW of Nördlingen, Bavaria. They were described as a Mesolithic burial.

Analysis of the cranium Ofnet 27 showed: $F = 0.05\%$, $100F/P_2O_5 = 0.24$, e $U_3O_8 = 2$ ppm, $N = $ nil.

[7]determined as e U_3O_8

Table 10   Analyses, Germany, early Middle Pleistocene. All specimens in the collection of Geol. Pal. Inst. Univ., Heidelberg.

BM(NH) Lab. No.	Locus and Description	CFH No.	F%	P_2O_5%	$\dfrac{F\%}{P_2O_5\%} \times 100$	$CaCO_3$%	N%	$e\,U_3O_8$ ppm	U ppm
G. 1	Mauer sand, *Homo heidelbergensis* mandible	M1	1·13	11·5	10	48	0·08!(w)	6	7
G. 2	Mauer sand, *Homo heidelbergensis* premolar dentine	M1	4·2	31·4	13	—	—	—	—
G. 3	Mauer sand, *Palaeoloxodon* skull	—	2·11	31·8	6·6	9·1	0·12(w)	11	—
G. 4	Mauer sand, *Palaeoloxodon* tusk dentine	—	1·67	35·0	4·8	7·6	nil	15	—
G. 6	Mauer sand, *Palaeoloxodon* tusk dentine	—	2·36	33·4	7·1	9·1	0·07	—	—
G. 5	Mauer sand, *Dicerorhinus etruscus* molar dentine	—	3·06	34·0	9·0	10·7	nil	—	—
G. 7	Mauer sand, *Dicerorhinus etruscus* mandible	—	2·21	29·7	7·4	10·7	<0·01 (w)	15	18

Table 11   Analyses, Germany, late Middle Pleistocene. All specimens in the collection of Staatliches Mus. Natur., Stuttgart.

BM(NH) Lab. No.	Locus and Description	CFH No.	F%	P_2O_5%	$\dfrac{F\%}{P_2O_5\%} \times 100$	$CaCO_3$%	N%	$e\,U_3O_8$ ppm	U ppm	Register No.
G. 29	Steinheim Gravels, *Homo steinheimensis* cranium	S1	1·2	11·8	10·2	—	0·37 (w)	2	2·5	—
G. 9	Steinheim Gravels, *Bison* horn-core	—	1·88	31·6	5·9	14·7	—	—	—	—
G. 10	Steinheim Gravels, *Homotherium* maxilla	—	1·40	32·5	4·3	12·2	nil (w)	—	—	—
G. 12	Steinheim Gravels, *Palaeoloxodon* level, rolled bone	—	1·98	30·9	6·4	13·9	0·23	13	—	—
G. 13	Steinheim Gravels, *Mammuthus* tusk dentine	—	1·55	33·4	4·7	13·2	—	—	—	—
G. 31	Steinheim Gravels, 'Elephas' tusk dentine	—	1·46	22·4	6·5	9·6	—	—	—	17492
G. 32	Steinheim Gravels, 'Elephas' tusk dentine	—	1·10	19·9	5·5	11·7	—	—	—	17753
G. 33	Steinheim Gravels, 'Elephas' tusk dentine	—	2·20	31·4	7·0	13·8	—	—	—	19022
G. 34	Steinheim Gravels, 'Elephas' tusk dentine	—	1·51	24·3	6·2	11·0	—	—	—	18070b

The nitrogen determination 'nil' in fact means not detected, but the element is probably present although at less than 0·01%. As 51 cervical vertebrae are available in the Ofnet collection, further nitrogen estimations should be made. Early Holocene series of bones are known in which the nitrogen content varies from <0·01% to 3%. The aim of this additional work would be to find material containing sufficient collagen for reliable radiocarbon dating.

Collagen extracted from one of the Ofnet skulls is said to have been dated by ^{14}C as c. 13 000 BP (when Magdalenian culture prevailed). Judgement on this result must be suspended until details of the sample UCLA-1783 have been published with full comments in *Radiocarbon*. All the Ofnet skulls appear to have been buried contemporaneously, so the UCLA date contradicts the evidence of the geometric microliths associated with two of these crania, indicating a Mesolithic culture of less than 10 000 years before present.

References: Gieseler 1971 : 201–202, Glowatzki & Protsch 1973, Newell *et al.* 1979.

RHÜNDA (Hessen). On 20 July 1956, Herr Glatzer found a hominid skull at a depth of 0·8 m in argillaceous silts near the village of Rhünda in the Melsungen district of Hessen. It consists of the right half of a cranium with occipital bone and isolated molars and premolars. At first it was interpreted as a female *Homo neanderthalensis*, but full investigation showed it to be a skull of *Homo sapiens* dating from early Holocene times.

Homo cranium: F = 1·29%, 100F/P_2O_5 = 4·8, e U_3O_8 = 8 ppm, N = 1·34% (w)

No well-dated fossil bones from the locality were available to compare. Comparison with analyses of bones of known age from further afield suggested that the Rhünda skull was not older than Würmian. Measurement of ^{13}C and ^{14}C in carbonate forming a calc-sinter incrustation of the cranium indicated an age of c. 9000 bp (H. 571-981). The inferred stratigraphical age is Pre-Boreal/Boreal.

References: Gieseler 1971 : 203–204, Oakley 1958a.

STEINHEIM (Württemberg). On 24 July 1933 K. Sigrist found an adult hominid cranium in the Sigrist Gravel pit on the northern outskirts of Steinheim an der Murr, 20 km north of Stuttgart. F. Berckhemer recognized that it was from the *Palaeoloxodon antiquus* bed of gravel, which he regarded as, at latest, of Saalian interstadial age. Further investigation by K. D. Adam indicated that the probable stratigraphical age is Elster-Saale (= Mindel-Riss) interglacial.

Homo cranium: F = 1·2%, 100F/P_2O_5 = 10·16, e U_3O_8 = 2 ppm, N = 0·37% (w)
Mammalian bone from *Palaeoloxodon* gravel: F = 1·98%, 100F/P_2O_5 = 6·4, e U_3O_8 = 13 ppm, N = 0·23%
Homotherium ('*Epimachairodus*'): F = 1·40%, 100F/P_2O_5 = 4·3, N = nil (w).
See Table 11, p. 38.

References: Adam 1954, 1961, Berckhemer 1933, Gieseler 1971 : 206–207.

Hungary

SUBALYUK. In 1932 neandertalian skeletal material (1 adult, 1 child) was found at the cave known as Mussolini-barlang in the Bukk Mountains. The fluorine content of hominid and mammalian bones from this cave was determined as <0·1%.

Reference: Thoma & Vértes 1971 : 226–227.

VÉRTESSZÖLLŐS. On 21 August 1965, L. Skoflek and J. Futó, working under direction of L. Vértes, discovered a hominid occipital in lime-mud at the bottom of a calc-tufa basin exposed in the travertine quarry near Vértesszöllős, 50 km west of Budapest. The mammalian fauna from the same stratigraphical level indicates that the deposits belong to an Elsterian interstadial (i.e. inter-Mindel.) The associated Buda industry has Clactonian traits, but it could be referred either to the pebble-tool tradition, or to the chopper/chopping-tool tradition.

Hominid occipital (Vértesszöllős 2): $F = 1\cdot6\%$, $100F/P_2O_5 = 5\cdot6$, $e\ U_3O_8 = 3$ ppm, $N =$ nil, $CaCO_3 = 18\%$

Directly associated mammal bone, cultural horizon 1: $F = 1\cdot5\%$, $100F/P_2O_5 = 5\cdot3$, $e\ U_3O_8 = 3$ ppm, $N = 0\cdot1\%$, $CaCO_3 = 20\%$

Other mammal bones from Vértesszöllős:
Cultural horizon 1: $F = 1\cdot8\%$, $100F/P_2O_5 = 5\cdot5$, $e\ U_3O_8 = 9$ ppm, $N = 0\cdot1\%$, $CaCO_3 = 11\%$
Cultural horizon 2: $F = 2\cdot0\%$, $100F/P_2O_5 = 6\cdot6$, $e\ U_3O_8 = 9$ ppm, $N = 0\cdot1\%$, $CaCO_3 = 12\%$
Cultural horizon 2/3: $F = 1\cdot5\%$, $100F/P_2O_5 = 6\cdot4$, $e\ U_3O_8 = 9$ ppm, $N =$ nil, $CaCO_3 = 12\%$
Cultural horizon 3: $F = 1\cdot5\%$, $100F/P_2O_5 = 5\cdot1$, $e\ U_3O_8 = 18$ ppm, $N = 0\cdot1\%$, $CaCO_3 = 12\%$
Cultural horizon 3: $F = 1\cdot63\%$, $100F/P_2O_5 = 5\cdot0$, $e\ U_3O_8 = 14$ ppm, $N = 0\cdot1\%$, $CaCO_3 = 13\cdot2\%$
Loess overlying cultural horizon 3: $F = 1\cdot8\%$, $100F/P_2O_5 = 4\cdot3$, $e\ U_3O_8 = 9$ ppm, $N = <0\cdot1\%$, $CaCO_3 = 12\%$

References: Oakley 1966, Thoma & Vértes 1971 : 228–229.

Italy

CASTENEDOLO (Lombardia). In 1860, G. Ragazzoni discovered hominid skeletal fragments on the hill of Castenedolo near Brescia, but since there was doubt about their stratigraphical age they were discarded as of no importance. In 1880, G. Ragazzoni found close to the same site several hominid skulls, with some associated post-cranial bones, including an adult female calvaria (Castenedolo 1), fragments of parietal and occipital bones of an adult male (Castenedolo 2) and isolated cranial fragments of a child (Castenedolo 3). These remains were embedded in shelly marine clay identified as of Tertiary (Astian) age by G. B. Cacciamali (1896), who cautiously rejected the opinion of those who regarded the human bones as contemporaneous with the clay. In 1889 further human bones were found at this site, but A. Issel (1889) recognized them as recent burials. G. Sergi agreed with Issel's opinion about the 1889 finds, although he maintained his earlier view (Sergi 1884) that those of 1880 (Castenedolo 1, 2 and 3) constituted proof that man with modern morphology (*Homo sapiens*) existed in Pliocene times.

Castenedolo 1–3 are still encrusted with their original matrix, and with the co-operation of Professor G. Genna were re-investigated in 1965. Analyses of the bones showed that their residual collagen (assessed by percentage of nitrogen) is higher than that of any fossil bones from central and northern Italian sites which have been tested with the exception of a few from Upper Pleistocene levels.

	%N
Castenedolo 1, cranial fragment:	1·6
Castenedolo 1, costae:	2·6
Castenedolo 2, cranial fragment:	2·2
Castenedolo 3, cranial fragment:	1·2
Middle Pleistocene bones from Italian sites:	<0·1–0·3
Upper Pleistocene bones from Italian sites:	<0·1–3·7
Holocene bones from Italian sites:	0·5–4·4

From this evidence it was inferred that Castenedolo 1–3 were intrusive burials into the Astian clays. In 1969 the British Museum Research Laboratory undertook radiocarbon dating of bones of Castenedolo 1, which proved to be 958 ± 116 years bp (BM-496) on the basis of ^{14}C dating of residual collagen in its vertebrae and costae (Barker, Burleigh & Meeks 1971 : 183).

Radiometric assays of the Castenedolo bones indicated an unexpectedly high uranium content:

Castenedolo 1, cranial fragment: $e\ U_3O_8 = 29$ ppm
Castenedolo 1, costae: $e\ U_3O_8 = 32$ ppm
Castenedolo 2, cranial fragment: $e\ U_3O_8 = 17$ ppm
Castenedolo 3, cranial fragment: $e\ U_3O_8 = 9$ ppm

The uranium content of the matrix of the bones was measured by Dr T. K. Ball of the Institute of Geological Sciences. He reported $3\cdot9 \pm 0\cdot2$ ppm.

The most likely explanation of the radioactivity of the bones is that the local ground-water is

Table 12 Analyses, Hungary, Middle Pleistocene.

BM(NH) Lab. No.	Locus and Description	CFH No.	F%	P_2O_5%	$\frac{F\%}{P_2O_5\%} \times 100$	N%	$CaCO_3$%	U_3O_8 ppm	Collection & Register No.
H. 1	Vértesszöllős, *Homo* occipital bone	V2	1·6	28·4	5·6	nil	18	3	Magyar Nemzeti Muzeum Pb65/1264
H. 2	Vértesszöllős, mammal bone (associated with *Homo*) cultural horizon 1	—	1·5	29·1	5·3	0·1	20	3	Magyar Nemzeti Muzeum —
H. 3	Vértesszöllős, cultural hor. 1 animal bone	—	1·8	32·4	5·5	0·1	11	9	K. P. Oakley Coll. unreg.
H. 4	Vértesszöllős, cultural hor. 2 animal bone	—	2·0	30·4	6·6	0·1	12	9	K. P. Oakley Coll. unreg.
H. 5	Vértesszöllős, cultural hor. 2/3 calcreted animal bone	—	1·5	33·8	6·4	nil	12	9	K. P. Oakley Coll. unreg.
H. 6	Vértesszöllős, cultural hor. 3 animal bone	—	1·5	30·1	5·1	0·1	12	18	K. P. Oakley Coll. unreg.
H. 15	Vértesszöllős, cultural hor. 3 animal bone	—	1·63	32·4	5·03	—	13·2	14	K. P. Oakley Coll. unreg.
H. 7	Vértesszöllős, Late Mindelian loess overlying cultural hor. 3 animal bone	—	1·8	41·6	4·3	<0·1	12·4	9	K. P. Oakley Coll. unreg.
H. 8	Vértesszöllős, cult. hor. 1 *Cervus elaphus* enamel	—	1·9	32·4	5·8	0·1	12·5	9	K. P. Oakley Coll. unreg.
H. 9	Vértesszöllős, Mindelian interstadial calcareous deposit ungulate bone	—	2·02	33·9	6·0	0·1	11·6	15	L. Vértes Coll., BM(NH) E.6190
H. 10	Vértesszöllős, Mindelian interstadial calcareous deposit animal bone	—	1·33	34·0	3·9	0·1	11·9	7	L. Vértes Coll., BM(NH) unreg.

rich in uranyl ions. Professor E. Anati of the Centro Camuno di Studi Preistorici, Brescia, sent a sample of well water from Castenedolo to the Centro di Studi Nucleari E. Fermi in Milan, where Professor Terrani determined 180 ml contained 1.58 ± 0.39 micrograms of uranium. As this is an unusually high proportion of uranium for ground-water, it probably accounts for accumulation of this element in the bones to the levels recorded after an exposure of less than 1000 years.

The fluorine content of the Castenedolo bones is also high for bones of late Holocene age. The Laboratory of the Government Chemist, London, could not detect fluorine in a sample of well water from Castenedolo, but in this tectonically rather unstable region the composition of ground-water is probably liable to considerable variation in the course of a millennium. The fluorine content of the Castenedolo bones is occasionally exceeded by that of bones of comparable age in other parts of Europe, for example County Durham:

Castenedolo bones: $F = 0.4–0.6\%$, $100F/P_2O_5 = 1.4–2.3$
Human bone from Mediaeval site, Barnard Castle, Co. Durham: $F = 1.3\%$, $100F/P_2O_5 = 4.1$

References: Barker, Burleigh & Meeks 1971 : 183, Cacciamali 1896, Issel 1889, Oakley *in* Sergi, Cardini & Leonardi 1971 : 235–236, Ragazzoni 1880, Sergi 1884.

CIRCEO (Latina). The neandertal skull Circeo 1 was found in 1939 occupying a hollow in the surface of an indurated cave filling formed by beach and sandy continental deposits in Grotta Guattari, 300 m SE of San Felice Circeo, Latina. Partial analysis of a nasal bone of the skull showed: $F = 0.57\%$, $100F/P_2O_5 = 5.5$, $N = 0.28\%$, $CaCO_3 = 36.1\%$.

The carbonate content is unusually high. In Pleistocene bones from other Italian sites $CaCO_3$ ranged from 3% to 27%. The exceptionally high carbonate content of this skull is presumably due to the macropores of the bone containing microcrystals of calcite or aragonite deposited by the stalagmite-forming water in this cave. In future research a thin section of the skull bone should be prepared for examination under a petrographic microscope.

The neandertal mandible Circeo 3 from breccia at the entrance to Grotta Guattari was tested for nitrogen representing residual collagen: $N < 0.1\%$ (w).

Reference: Sergi, Cardini & Leonardi 1971 : 237–239.

OLMO (Tuscany). I. Cocchi obtained in 1863 a calotte of *Homo* from the cutting of the Florence–Rome railway at Olmo near Arezzo. The cutting exposed Pleistocene gravels overlain by clay, but the layer of origin of the human skull fragment was uncertain. Analytical dating at the BM(NH) indicated that the calotte probably came from the gravel, which contained a blade of Upper Palaeolithic type.

Homo calotte: $F = 0.13\%$, $100F/P_2O_5 = 2.5$, $N = 3.32\%$
Equus bones, probably from gravel: $F = 0.3\%$, $100F/P_2O_5 = 1.3$, $N = 2.64\%$
Elephas dentine, probably from underlying clay: $F = 2.3\%$, $100F/P_2O_5 = 7.5$, $N = 0.92\%$

Reference: Sergi, Cardini & Leonardi 1971 : 247–248.

POFI (Rome). In November 1959 a neandertal ulna was found in rearranged pozzolana underlying lacustrine deposits with diatomites in a quarry 6 km from Pofi, SSE of Frosinone, Lazio, Rome. The containing deposit is probably Riss–Würm in age. Analysis of the bone showed: $F = 1.5\%$, $100F/P_2O_5 = 6.4$, $e\ U_3O_8 = 9$ ppm, $N = 0.2\%$. See Table 13, p. 44.

Reference: Sergi, Cardini & Leonardi 1971 : 249–250.

QUINZANO (Verona). In 1938 workmen directed by G. Montresor found a human occipital fragment in alluvial deposits within Cava Vecchia, exposed in a clay pit at Ca'Rotta Quinzano, near Verona. According to Montresor the human fragment came from the base of layer III (Upper Palaeolithic?), but the degree of mineralization, colour and limestone caking suggested to R. Battaglia that it had been derived from a much older layer.

Analytical dating carried out by the BM(NH) gave results which supported the view that the occipital fragment antedated layers III-V (Würm), and was probably derivative from layers VI/VII.

Homo occipital (Quinzano 1): F = 0·75%, 100F/P_2O_5 = 3·9, *e* U_3O_8 = 11 ppm, N = 0·3% (w)
Equus hydruntinus dentine, layer III: F = 0·16%, 100F/P_2O_5 = 0·6, *e* U_3O_8 = nil, N = 1·46% (w)
Mammal bones from layer V: F = 0·4–1·1%, 100F/P_2O_5 = 2·5–6·6, N = 0·11 (w)
Antler, bone and dentine from layers VI/VII: F = 0·5–1·5%, 100F/P_2O_5 = 4·4–6·6, *e* U_3O_8 = 31 ppm,
N = 0·33%
See Table 13, p. 44.

Layer VII, the oldest layer, contained flakes of Clactonian facies and biface of 'Chellean' type (= Acheulian). Layer VI contained a molar of *Mammuthus trogontherii intermedius*. A. Pasa correlated VI with Riss–Würm interglacial and VII with Riss, but differentiation of material from these two layers was not clear.

Reference: Sergi, Cardini & Leonardi 1971 : 250–251.

SACCOPASTORE (Rome). Two neandertal skulls were found in fluviatile gravel forming the lowest terrace of the River Aniene exposed in a gravel pit at Saccopastore, 3·5 km from Porta Pia, Rome. Saccopastore 1 was found on 13 May 1929 by S. Sergi; Saccopastore 2 on 16 July 1935 by A. C. Blanc and H. Breuil. In chemical composition the bone of these skulls conforms with that of associated *Hippopotamus* dentine.

Saccopastore 1: F = 1·8%, 100F/P_2O_5 = 8·1, N = 0·14%
Saccopastore 2: F = 1·7%, 100F/P_2O_5 = 8·3, N = 0·12%
Hippopotamus dentine: F = 1·6%, 100F/P_2O_5 = 5·6, *e* U_3O_8 = 52 ppm, N = 0·16%
See Table 13, p. 44.

The samples of human bone were inadequate for radiometric analysis, but if needed their uranium content could be determined by microchemical analysis.

The terrace gravel is attributed to the Riss–Würm interglacial. The radiocarbon age of *Abies* wood from a correlated deposit in the Mussolino channel near Latina has been reported as 58 000 ± 500 yrs bp (GrN-2572).

Reference: Sergi, Cardini & Leonardi 1971 : 254–255.

SEDIA DEL DIAVOLO (Rome). In 1956 a femur fragment and a metatarsal of a hominid were found in Nomentanan (= Riss) gravels at Sedia del Diavolo quarry overlooking the river Aniene, about 3·5 km from Porta Pia, Rome. Analyses showed:

femur: F = 1·83%, 100F/P_2O_5 = 6·64, N = nil
metatarsal: N = 0·19% (w)

Reference: Sergi, Cardini & Leonardi 1971 : 257–258.

Malta

Two taurodont molar teeth of *Homo* were found in Ghar Dalam Cave in 1917, associated in broad sense with fossil bones of *Cervus*. Sir Arthur Keith referred the teeth to *Homo neanderthalensis* on the basis of their taurodontism. Although obsidian artefacts of Neolithic type occurred in the cave-earth with the teeth, it was suggested that the latter might have been derived from a much older deposit in the cave, which had been partly filled in Pleistocene times. During a visit to Malta in 1962 I obtained permission from the Director of the National Museum to take samples of the Ghar Dalam specimens (including one of the taurodont teeth) for nitrogen analysis in London. The results were as follows:

Ghar Dalam 2 (r. M^3) dentine: N = 1·85%
Cervus long bone from Red Earth, Late Pleistocene or Early Holocene: N = 0·13%
Hippopotamus dentine from Pleistocene Bone Breccia: N = nil.
Dentine of teeth from various Neolithic sites, Malta: >0·3%

These analyses indicated that Ghar Dalam 2 is not older than Neolithic. In 1936, another taurodont molar (l. M_3) was found by Dr J. G. Baldacchino in black clayey earth containing artefacts of the first or Ghar Dalam phase of the Maltese Neolithic.

Table 13   Analyses, Italy, early Upper Pleistocene.

BM(NH) Lab. No.	Locus and Description	CFH No.	F%	P_2O_5%	$\dfrac{F\%}{P_2O_5\%} \times 100$	$CaCO_3$%	N%	$e\,U_3O_8$ ppm	Collection
It. 30	Cava Pompi, Pofi str I (with *Palaeoloxodon*), *Homo* tibia	P1	1·48	23·3	6·4	3·3	0·18	9	L. Cardini Coll. Museo della Preistoria del Lazio, Rome
It. 6	Cava Vecchi, Quinzano str VI, *Mammuthus trogontherii* bone	—	0·5	11·5	4·4	—	—	—	Museo Storia Naturale, Verona
It. 8	Cava Vecchi, Quinzano str VI, '*Elephas*' tusk dentine	—	1·5	33	4·5	—	0·33 (w)	—	Museo Storia Naturale, Verona
It. 9	Cava Vecchi, Quinzano str VI, '*Elephas*' bone	—	1·2	27·5	4·4	—	—	—	Museo Storia Naturale, Verona
It. 10	Cava Vecchi, Quinzano str VII, *Megaloceros* antler	—	0·35	4·0	8·7	—	—	—	Museo Storia Naturale, Verona
It. 11	Cava Vecchi, Quinzano str VII, cervid bone	—	0·6	9·0	6·6	—	0·11 (w)	31	Museo Storia Naturale, Verona
It. 28	Low terrace gravel Aniene, Saccopastore, Rome, *Hippopotamus* dentine	—	1·61	28·7	5·6	2·9	0·16	52	Istituto di Antropologia, University of Rome
It. 29	Low terrace gravel Aniene, Saccopastore, Rome, *Hippopotamus* bone	—	—	—	—	—	0·13	50	Istituto di Antropologia, University of Rome
It. 31	Low terrace gravel Aniene, Saccopastore, Rome, *Homo* cranium	S1	1·76	21·7	8·1	15·3	0·14	—	Istituto di Antropologia, University of Rome
It. 33	Low terrace gravel Aniene, Saccopastore, Rome, *Homo* cranium	S2	1·67	20·0	8·3	26·6	0·12	—	Istituto di Antropologia, University of Rome

The dental surgeon Dr J. J. Mangion of the Royal University of Malta recorded in 1962 that he had extracted taurodont teeth from the jaws of two modern Maltese patients.

References: Keith 1925, Mangion 1962, Oakley 1971*b*.

Netherlands

BEEGDEN. A dozen or so hominid bones (cranial and post-cranial) were salvaged between 1962 and 1966 from material dredged from the gravel bed of the river Meuse at Beegden, south of Roermond, Limburg. According to C. J. Overweel, when the X-ray powder-diffraction method was applied, the fluorine content of two of these bones (Beegden 1 and 2) showed close agreement with dentine of *Mammuthus* dredged from river gravel and sand in the same region. This may be of late Weichselian age. However, analyses of nine of the Beegden human bones showed that they had such a high collagen content (measured as %N) that unquestionably they are not of great antiquity and are certainly post-Pleistocene.

Beegden 5: N = 3·91%
Beegden 6: N = 4·35%
Beegden 7: N = 4·43%
Beegden 8: N = 4·17%
Beegden 9: N = 4·04%
Beegden 10: N = 3·04%
Beegden 11: N = 4·26%
Beegden 12: N = 3·88%
Beegden 13: N = 3·53%

References: Erdbrink & Overweel 1971 : 226–267, Erdbrink, Meiklejohn & Tacoma 1975, Newell *et al.* 1979 : 181.

HENGELO. Two human calottes were found in yellowish brown sand and dark grey marl during harbour excavations *c.* 2 km south of the railway station at Hengelo, Overijssel, in 1934–35. Although Hengelo 1 was originally believed to be of Upper Pleistocene age, both calottes are now considered to be Early Holocene (Mesolithic). It may be regarded as the first authenticated find in the series known as the 'River Valley' people, who occupied a large area of the Rhine delta and surrounding plains, from Norfolk (e.g. Strumpshaw), England, to Téviec, France. They had an industry of somewhat Maglemosian aspect.

According to J. Butter (1952, *in lit.*) analysis of Hengelo 1 showed F = 0·28–0·30%.

References: Erdbrink & Overweel 1971 : 267–268, Erdbrink, Meiklejohn & Tacoma 1975, Newell *et al.* 1979 : 182.

Russia

KIIK-KOBA (Crimea). Although no material from this limestone cave was investigated at the BM(NH), for comparative purposes it is worth recording here that samples of bone from this Mousterian site were analysed by V. V. Danilova.

Kiik-Koba 1 (*Homo neanderthalensis*): F = 0·36%, $100F/P_2O_5$ = 0·12
Saiga tatarica: F = 0·7%, $100F/P_2O_5$ = 0·22–0·3

References: Danilova 1946, Klein, Ivanova & Debetz 1971 : 318–319.

Spain

BARRANC BLANC. In 1951, a hominid frontal bone was discovered by M. Jorda working with J. & P. Cubero Garcia in an Epigravettian occupation layer in cave-earths at Barranc Blanc, a cave near Rótova, about 10 km from Gandia, Valencia. In 1953, the cranium of a young adult hominid was found at approximately the same archaeological horizon. Analyses of these hominid

bones indicated that their nitrogen content was sufficiently low (0·15–0·38%) to be consistent with an Upper Palaeolithic age. The e U_3O_8 content proved to be nil detected.

Reference: Garralda & Irwin 1971 : 288–289.

CARIGÜELA. In 1955, J.-C. Spahni discovered a series of hominid bones at two occupation levels in the cave-earths at Carigüela, the cave 1 km east of the village of Pinar, Granada. At levels 6–7 the hominids (Carigüela 1–3) were associated with a typical Mousterian industry; at level 2 the hominids (Carigüela 4–6) were accompanied by a Mousterian industry showing slight Aurignacian influences.

Samples of the hominid bones (and one associated animal bone from level 6) were analysed. They all showed a low fluorine content (c. 0·1%), but this is commonly the case with bones from cave deposits. The protein residue measured as N% had been reduced to a very low or negligible level as was to be expected. The e U_3O_8 content proved to be nil detected.

Reference: Garralda & Irwin 1971 : 290–291.

CÒVA NEGRA. In 1933, a neandertalian parietal bone was found by P. Gonzalo Vines in a Mousterian occupation layer in the cave of Nova Negra, near the village of Játiva, Valencia.

Analyses of the bone showed a very low fluorine content (0·08%) but the protein residue (measured as nitrogen) had been reduced to a level consistent with Upper Pleistocene age. The e U_3O_8 content proved to be nil detected.

Reference: Garralda & Irwin 1971 : 292–293.

PARPALLÓ. In 1930, L. Pericot Garcia discovered the skull of a hominid child at a Solutrean occupation level in the cave of Parpalló, 10 km NW of the village of Candia, Valencia.

Analyses showed an exceptionally low level of fluorine (0·06%) but the protein residue had been reduced to 0·3% N, which is consistent with Late Pleistocene antiquity. The e U_3O_8 content proved to be nil detected.

Reference: Garralda & Irwin 1971 : 295.

Switzerland

BIRSMATTEN (Berne). In 1944, Carl Lüdin discovered the greater part of a human skeleton with skull in argillaceous deposits on the floor of the Birsmatten rock-shelter, overlooking the River Birse in the Laufen district. Mesolithic industries (at level D resembling Sauveterrian, at level E resembling Tardenoisian) occurred in these sediments. The skull and skeleton clearly represented a penecontemporaneous burial, but it is uncertain whether it is contemporary with material of level D or level E.

Birsmatten 1: N = 1·1%
Animal bone level D: N = 1·5–2·1%
Animal bone level E: N = 1·6%
Radiocarbon dating of animal bones gave the following results:
level D = 6970 ± 120 BP (B-1236)
level E = 7670 ± 120 BP (B-237)

Level D (III) has been correlated by pollen as of Boreal age, and level E (II) as belonging to the Boreal/Atlantic transition.

References: Oakley 1964a, Sauter 1971a : 304–305.

LE SCÉ (Vaud). In 1868–69, fragmentary post-cranial bones of several individuals (*Homo sapiens*) were found in Late Pleistocene and Early Holocene bone-breccias in the rock-shelter known as Le Scé du Château at Villeneuve in the Aigle district.

Table 14 Analyses, Spain, Upper Pleistocene.

BM(NH) Lab. No.	Locus and Description	CFH No.	F%	P_2O_5%	$\frac{F\%}{P_2O_5\%} \times 100$	$CaCO_3$%	N%	$e\,U_3O_8$ ppm	Collection and Register Number
SP. 43	Barranc Blanc cave, Solutrean, *Homo* frontal	BB1	—	—	—	—	0·38 (w)	nil	Museo del Servicio de Investigaciones Prehistoricas, Valencia Cr. 91
SP. 44	Barranc Blanc cave, Epigravettian, *Homo* calotte	BB2	—	—	—	—	0·15 (w)	nil	Museo del Servicio de Investigaciones Prehistoricas, Valencia, Cr. 117
SP. 31	Cariguela cave, occ. level 2, *Homo sapiens* mandible	C4	—	—	—	—	0·23 (w)	nil	Museo Arquelogico Provincial de Granada, 5.873
SP. 32	Cariguela cave, occ. level 2, *Homo sapiens* parietal	C5	0·17	26·5	0·6	11·1	2·2 (w)	nil	Museo Arquelogico Provincial de Granada, 5.874
SP. 33	Cariguela cave, occ. level 2, *Homo sapiens* tibia	C6	—	—	—	—	1·3 (w)	nil	Museo Arquelogico Provincial de Granada, 5.878
SP. 37	Cariguela cave, occ. level 2, *Cervus elaphus* mandible	—	0·04	21·7	0·2	20·5	—	nil	Museo Arquelogico Provincial de Granada
SP. 38	Cariguela cave, occ. level 3, *Panthera spelaea* humerus	—	—	—	—	—	0·25 (w)	nil	Museo Arquelogico Provincial de Granada
SP. 34	Cariguela cave, occ. level 6, *Homo neanderthalensis* parietal	C3	0·1	20·8	0·5	21·3	0·16	nil	Museo Arquelogico Provincial de Granada, 5.875
SP. 39	Cariguela cave, occ. level 6, *Cervus elaphus*	—	0·17	26·7	0·6	24·2	nil	nil	Museo Arquelogico Provincial de Granada
SP. 35	Cariguela cave, occ. level 7, *Homo neanderthalensis* parietal	C1	0·08	21·0	0·4	23·1	nil	nil	Museo Arquelogico Provincial de Granada, 5.876
SP. 36	Cariguela cave, occ. level 7, *Homo neanderthalensis* frontal	C2	—	—	—	—	nil	nil	Museo Arquelogico Provincial de Granada, 5.877
SP. 40	Cariguela cave, occ. level 7, *Dicerorhinus mercki* skull	—	—	—	—	—	0·37 (w)	nil	Museo Arquelogico Provincial de Granada
SP. 41	Cova Negra cave, *Homo neanderthalensis* parietal	CN1	0·08	28·5	0·3	9·1	0·25 (w)	nil	Museo del Servicio de Investigaciones Prehistoricas, Valencia, Cr. 92
SP. 42	Parapalló cave, *Homo sapiens* occipital	P1	0·06	10·8	0·6	0·5	0·31 (w)	nil	Museo del Servicio de Investigaciones Prehistoricas, Valencia, Cr. 90

Human femur ('Scé III'): N = 1·74% (w)
Human bone (unspecified): N = 4·06% (w)
Rangifer long-bone, Late Pleistocene: N = 2·59% (w)

Reference: Sauter 1971*a* : 306–307.

Yugoslavia[8]

KRAPINA, Croatia. Neandertal hominid bones were excavated by K. D. Gorjanović-Kramberger in 1899 (and subsequent years) at the rock-shelter of Krapina on the right bank of the River Krapinica, NNW of Zagreb, Croatia. The hominid bones, representing about 25 individuals (including five children) were embedded in sandy loams derived from weathering débris of the Miocene sandstone in which the shelter had been cut. The shelter deposits are underlain by fluviatile sand and gravel.

In 1967, aided by a grant from the Wenner-Gren Foundation, Miss T. I. Molleson and Dr J. C. Vogel visited Krapina, and other bone-bearing sites in Yugoslavia, with the aim of collecting samples for relative dating and for radiocarbon measurement. They obtained a series of samples from the Krapina hominid and animal bones preserved in the Geološko-Paleontološki Muzej, Zagreb, by kind permission of the Director, Professor I. Crnolatac. They were unable to collect any field samples at Krapina, because the rock-shelter deposits have been totally removed—not even a witness section has been left at the site.

It is of historic interest that a hominid bone from Krapina was analysed for fluorine in preparation for the report on the original excavations (Gorjanović-Kramberger 1901). The value recorded was 0·7%.

Bone samples from most Mousterian levels in the shelter deposits were analysed in the Laboratory of the Government Chemist for fluorine and phosphate following the 1967 expedition, but no trend was detectable in the fluorine/phosphate ratio. However, nor was there any inconsistency between the ratio found in the hominid fragments and in the animal bones of the level with which they were reputed to be associated. These results may be summarized as follows:

Human bones (7): F = 0·73–1·04%, $100F/P_2O_5$ = 2·6–4·1
Animal bones (11): F = 0·22–1·53%, $100F/P_2O_5$ = 0·7–5·6

A series of hominid and animal bones samples from Krapina were submitted to radiometric assay in the BM(NH) and the results are summarized in Table 15. The counts obtained on the Krapina samples fall within the range for the Upper Pleistocene in Yugoslavia. No significance

Table 15   Uranium in bones from Mousterian layers at
Krapina. e U_3O_8 ppm

Human bone, Mousterian layer 9	nil
Animal bone, Mousterian layer 8	11
Animal bone, Mousterian layer 8	6
Animal bone, Mousterian layer 8	4
Animal bone, Mousterian layer 8	8
Animal bone, Mousterian layer 7	8
Animal bone, Mousterian layer 5	7
Animal bone, Mousterian layer 4	nil
Animal bone, Mousterian layer 4	nil
Human bone, Mousterian layer 4	6
Human bone, Mousterian layer 4	nil
Human bone, Mousterian layer 3	4
Human bone, Mousterian layer 3	5
Animal bone, Mousterian layer 1–2	10
Animal bone, Mousterian layer 1	10

[8]This section was prepared largely on the basis of a script written by Miss T. I. Molleson after her visit to Yugoslavia in September, 1967.

Table 16 Analyses, Yugoslavia, Upper Pleistocene. All specimens in Geološko-Paleontološki Muzej, Zagreb.

BM(NH) Lab. No.	Locus and Description	F%	P_2O_5%	$\dfrac{F\%}{P_2O_5\%} \times 100$	$CaCO_3$%	N%	$e\ U_3O_8$ ppm
Y.1	Krapina layer 9 *Dicerorhinus* bone	0·39	32·0	1·2	11·7	0·61 (w)	—
Y.2	Krapina layer 9 *Homo* femur	0·88	31·3	2·8	7·7	0·36 (w)	9
Y.3	Krapina layer 9 *Bos* mandible	0·73	29·9	2·4	8·4	0·78 (w)	11
Y.4	Krapina layer 9 *Dicerorhinus* bone	—	—	—	—	0·30 (w)	—
Y.5	Krapina layer 1 *Castor* bone	1·53	27·2	5·63	9·5	0·27 (w)	10
Y.6	Krapina layer 1–2 animal bone	0·61	26·8	2·28	11·2	0·57 (w)	10
Y.7	Krapina layer 3 *Homo* adult skull	0·97	24·3	3·99	10·0	nil	5
Y.8	Krapina layer 3 *Homo* juvenile skull	1·0	26·9	3·72	12·3	0·24 (w)	4
Y.9	Krapina layer 4 *Homo* adult skull	1·01	24·5	4·12	11·5	0·36 (w)	nil
Y.10	Krapina layer 4 *Homo* skull	1·04	37·5	2·7	11·7	0·31 (w)	nil
Y.11	Krapina layer 4 animal bone	1·28	28·4	4·5	8·6	0·42 (w)	6
Y.12	Krapina layer 4 *Homo* skull	1·09	25·2	4·3	6·0	0·30 (w)	7
Y.13	Krapina layer 5 animal bone	0·98	29·2	3·4	8·8	0·43 (w)	8
Y.14	Krapina layer 6a (7) animal bone	0·95	29·9	3·19	12·9	0·43 (w)	8
Y.15	Krapina layer 8 animal bone	0·77	29·2	2·64	6·1	0·51 (w)	4
Y.16	Krapina layer 6b (8) animal bone	0·88	27·6	3·20	7·6	0·70 (w)	11
Y.17	Krapina layer 8 animal bone	0·22	31·2	0·70	15·4	0·23 (w)	6
Y.18	Krapina layer 8 *Dicerorhinus* bone	0·68	28·7	2·37	14·3	0·35 (w)	nil
Y.19	Krapina layer 9g *Homo* nasion	0·73	27·9	2·60	13·4	0·14	6
Y.20	Krapina *Homo* skull[9]	0·83	29·9	2·77	14·2	0·08	5

[9]K. D. Gorjanović-Kramberger Coll. 1901. Geološko-Palaeontološki Muzej, Zagreb '4058–4059'.

Table 17   Analyses, Yugoslavia, Upper Pleistocene. All specimens in the M. Malez Coll., Geološko-Paleontolška laboratorij, Zagreb.

BM(NH) Lab. No.	Locus and Description	F%	P_2O_5%	$\frac{F\%}{P_2O_5\%} \times 100$	$CaCO_3$%	N%	$e\ U_3O_8$ ppm	^{14}C yrs bp
Y. 21	Šandalja Cave Upper Gravettian layer b, *Homo* calotte	—	—	—	—	0·78	—	12 320±100 (GrN-4978)[10]
Y. 22	Šandalja Cave Upper Gravettian layer b, *Homo* calotte	n.d.	32·1	0	12·3	—	—	10 830±50[10] (GrN-4976)
Y. 23	Šandalja Cave Upper Gravettian layer b, animal bone	0·03	32·7	0·09	20·1	0·27 (w)	nil	—
Y. 24	Šandalja Cave Lower Gravettian layer c, animal bone	0·22	28·9	0·75	16·4	1·51 (w)	nil	23 450±180 (GrN-5013)
Y. 25	Šandalja Cave Aurignacian layer e, animal bone	0·13	31·7	0·42	13·3	0·93 (w)	nil	25 340±170 (GrN-4977)
Y. 26	Šandalja Cave Aurignacian layer f, animal bone	0·18	28·2	0·62	14·5	1·67 (w)	nil	—
Y. 27	Šandalja Cave Upper Gravettian layer b (above hearth), animal bone	n.d.	29·0	0	13·0	1·93 (w)	nil	—
Y. 28	Šandalja Cave Upper Gravettian layer b (just above hearth), animal bone	0·05	32·8	0·14	10·0	1·57 (w)	nil	—
Y. 29	Šandalja Cave Aurignacian (?) layer c–d (below hearth), animal bone	0·08	29·5	0·27	15·3	0·55 (w)	5	—
Y. 32	Crvena Stijena, Montenegro, Upper Palaeolithic layer 10/11, animal bone	0·59	31·7	1·87	17·6	0·79 (w)	3	—
Y. 33	Crvena Stijena, Mousterian layer 12, animal bone	0·22	31·2	0·71	14·3	1·37 (w)	5	40 770±900 (GrN-6083)
Y. 34	Crvena Stijena, Mousterian layer 12, animal bone	0·30	28·9	1·02	11·9	1·68 (w)	7	>46 250 (GrN-4988)

[10]Associated charcoal.

can at this stage be attached to the very low counts for some bones in Mousterian layers 4 and 9. None of the hominid bone fragments contained more than 1·0 % nitrogen derived from residual collagen, as was to be expected with Upper Pleistocene material.

Dr J. C. Vogel measured the radiocarbon in collagenous residue from a sample of bone from Mousterian level 1, but the result he obtained, 3200 ± 800 bp (GrN-4983) indicated without any doubt that there had been contamination with recent carbon. He also measured the radiocarbon in a piece of charred bone from a Mousterian level at Krapina that had been obtained by Professor J. S. Weiner when he visited the Geološko-Paleontološki Muzej, Zagreb in 1962. The result was 30 700 ± 750 bp (GrN-4299). Bone from a Mousterian level at Crvena Stijena, Montenegro had a radiocarbon age of 40 770 ± 900 bp (GrN-6083), but there is a growing body of evidence that Mousterian culture survived here and there until at least 32 000 bp (see Mellars 1970; Molleson, Oakley & Vogel 1972).

References: Gorjanović-Kramberger 1901, Malez 1970; 1971 : 338–340, Mellars 1970, Molleson, Oakley & Vogel 1972.

ŠANDALJA, Istria. In 1963 and 1966, M. Malez discovered hominid fragments in a Gravettian occupation level (layer b) in cave deposits exposed in the chalk quarry at Šandalja in southern Istria.

The fluorine content of a hominid fragment found in 1963 was not detectable, but the range of fluorine shown by animal bones from the Upper Palaeolithic levels in this cave is also very low. The nitrogen content of the same bone is within the range shown by animal bones from the Gravettian layers (0·27–1·51 %).

Reference: Malez 1971 : 341–342.

VELIKA PEĆINA, Croatia. In 1961, M. Malez found a human frontal bone (*Homo* aff. *neanderthalensis*) in association with Proto-Aurignacian industry in layer j in the Velika Pećina cave, NW of Ivanec in Croatia. The nitrogen of this bone is 0·77 % (w). The inferred date of this hominid is > 33 850 ± 520 bp on the basis of ^{14}C dating of charcoal in the overlying layer i.

Reference: Malez 1971 : 342.

Concluding summary with Acknowledgements

Researches on the use of analytical techniques to aid the relative dating of fossil hominid and other skeletal materials have been pursued by the present writer since the end of World War II. In April 1947 I returned from an official visit to East Africa with a collection of fossil bones and teeth, obtained mainly through the co-operation of the late Dr L. S. B. Leakey, from some of the classic hominoid sites in Kenya and Tanganyika (now Tanzania). I was fortunate enough to obtain later that year an offer from Dr H. J. Walls, then Director of the Forensic Science Laboratory, Bristol, to test the fluorine-dating method by analysing a selected series of the fossil specimens that I had brought back from Africa, and which ranged in age from Miocene to Early Holocene (although the majority were Pleistocene). The results showed that Carnot's method was not very dependable when applied to skeletal material from regions where the ground-water is rich in fluorine, as in sediments containing a high proportion of volcanic ash. In such situations the apatite composing buried bones and teeth rapidly approaches mineralogical saturation with fluorine. Under tropical conditions this process is not only accelerated but occurs in a more random fashion. The fluorine content of bones is far from being uniform in any single stratum in the Lower and Middle Pleistocene succession of ashy deposits exposed in the Olduvai Gorge, Tanzania. Some of the bones in the Late Pleistocene pyroclastic deposits at Kuguta, near Homa Mountain, Kenya, have a fluorine content in excess of the maximum for fluorapatite (see p. 3).

As the majority of specimens analysed for fluorine by Carnot were not from tropical/volcanic areas, it is probable that relative dating by fluorine is more reliable when applied to specimens buried in deposits where the ground-water has a low or moderate fluoride content, and where sedimentation and weathering occur under temperate climatic conditions.

In 1948, the late W. N. Edwards, then Keeper of Geology (Palaeontology) at the BM(NH), agreed to approach the Department of the Government Chemist (London) with a view to their undertaking microchemical analyses of bone, antler, dentine and enamel samples collected from Quaternary deposits in the Swanscombe district, Kent, from which several hominid specimens of disputed age had been recovered. The Government Chemist agreed to this co-operative programme of research. The first results were notably successful. Before the end of 1948 it was shown that the controversial Galley Hill skeleton had a very low fluorine content and was therefore evidently intrusively buried into the 100-ft terrace of the Thames during Post-Pleistocene time. In marked contrast the Swanscombe skull showed the relatively high fluorine content which characterizes the Middle Pleistocene fauna contemporaneous with the gravels of the 100-ft terrace.

In this first demonstration in Europe since the nineteenth century of the usefulness of Carnot's test the analysts (R. H. Settle, E. C. W. Maycock and C. R. Hoskins) made the innovation of determining the phosphate as well as the fluorine content of the specimens, and thus found that the fluorine/phosphate ratios were better indicators of the relative ages of buried bones of varied origin than the fluorine percentages on their own.

The following year (1949) microsamples of the hominoid and 'associated' mammalian skeletal remains reported as having been found at Piltdown, near Lewes, Sussex, between c. 1910 and 1915, were submitted to the Department of the Government Chemist where they were analysed by Dr C. R. Hoskins. The results which he obtained were extraordinary: the hominoid bones and teeth from both Piltdown sites (Site I and Site II) had such a low fluorine content (in the 1949 results ranging from less than 0.1% to 0.4%) that comparison with the fluorine content of known Pleistocene skeletal remains from sites in southern Britain made it impossible to believe any longer that Piltdown man had the high antiquity which for some 40 years anthropologists and palaeontologists had been claiming. The fossil mammalian bones and teeth said to have been found at the two Piltdown sites showed a wide range of fluorine content, from less than 1% to 2.7%. This was not so surprising because several of the original investigators had concluded that fossil faunas of two or more geological ages had become mixed together in the Piltdown river gravel. It was so difficult to accept the new indications that the Piltdown skull was at the most no older than Upper Pleistocene, that further analytical investigations of the anomalous results of 1949 were set in motion using larger samples and more refined techniques of analysis. The outcome in 1953 was to establish Dr (later Professor) J. S. Weiner's hypothesis that the Piltdown I skull (including mandible and teeth) had been forged. Although it was the application of the 'fluorine test' to the Piltdown specimens in 1949 that triggered off the extensive investigations which eventually led to this result, the removal of all doubt about its correctness was due to teamwork on a scale probably unprecedented in solving a single problem. Thus a whole battery of physical and chemical techniques was brought to bear on the Piltdown problem before complete proof was obtained that the Piltdown skulls I and II and all the mammalian fossils recovered from Site I and Site II were part of an elaborate forgery. Some 25 scientists deserve credit for their contributions to this work: Dr G. F. (later Sir Frank) Claringbull, Mrs A. Foster, Dr M. H. Hey, Dr A. A. Moss and Dr J. D. H. Wiseman of the Department of Mineralogy, BM(NH); A. D. Baynes-Cope, H. L. Bolton, H. J. Dothie, C. F. M. Fryd and Dr C. R. Hoskins of the Department of the Government Chemist, London; Professor W. E. (later Sir Wilfrid) LeGros Clark and Dr J. S. Weiner,[11] Department of Human Anatomy, University of Oxford; Miss R. J. Plesters and Dr A. E. A. Werner, then at the Research Laboratory, National Gallery; Dr E. T. Hall, Clarendon Laboratory, Oxford; Dr R. C. Hoather, late of Counties Public Health Laboratories, London; Dr C. Bloomfield, Rothamsted Experimental Station, Harpenden; Dr A. V. W. Martin (now Mrs Angela Brown) and Professor J. T. Randall, Department of Biophysics, King's College, London; Drs G. Weiler and F. B. Strauss, Microanalytical Laboratory, Oxford; F. H. Edmunds, Geological Survey of Great Britain; S. H. U. Bowie and Dr C. F. Davidson[12], Atomic Energy Division, Geological Survey of Great Britain.

[11]Later Professor of Environmental Physiology, London University.
[12]Later Professor of Geology, University of St Andrews.

As the Piltdown investigations were directed from the Department of Geology (Palaeontology), BM(NH), I would like to record my appreciation of the co-operation received from the members of the staff of the Department who made fossils available for comparative tests, notably the late Dr A. T. Hopwood who at the time was in charge of Fossil Mammalia. L. E. Parsons and other members of the laboratory staff in the Department of Geology (Palaeontology) rendered valuable service in the preparation of samples for analysis. I should also like to record the helpfulness of the consultations which I had with the late Dr F. C. Fraser, eventually Keeper of the Department of Zoology. The Director of the Museum at the time, the late Sir Gavin de Beer, gave unreserved encouragement to all this work and played a leading part in presenting a summary of the results at a meeting of the Geological Society of London on the 30th June 1954.

Following the reports of successful applications of analytical techniques for the relative dating of fossil hominids, the late Dr Paul Fejos, Director of Research of the Wenner-Gren Foundation, told me after I had lectured on the subject in New York in 1950 that if I reached a position at which my researches on these lines would benefit by receiving a grant-in-aid, I should let him know and he would submit an application on my behalf for consideration by his Board of Directors. So far the researches had been on a fairly modest scale, but after the publication of the Piltdown investigations, W. N. Edwards agreed that as the Laboratory of the Government Chemist was prepared to continue the co-operation with the Museum, the research should be extended to a wide range of sites in Britain and abroad. Since some of the work would involve foreign travel, employment and purchase of equipment for use in the Museum, he thought that it would be a good idea for me to apply to the Wenner-Gren Foundation for grants-in-aid of the researches. My applications were successful.

The first grant was made in 1952, and was mainly used for the purpose of applying relative dating techniques to fossil hominid sites in southern Africa. After carrying out this programme of work in 1953, the remainder of the grant was devoted to continuing the use and development of analytical methods of dating to fossil hominids and associated faunal material from American, Asiatic and European sites. With a second grant received in December 1954, it was possible to purchase radiometric equipment to aid this work. I should like to record my gratitude to the late Dr Paul Fejos for the interest he took in these researches up to the time of his death in 1963. Mrs Lita Osmundsen (formerly Mrs Fejos) succeeded her late husband as Director of Research of the Wenner-Gren Foundation and continued to give me much help and encouragement in subsequent years. The Board of Directors in New York renewed their grant-in-aid of analytical work on fossil bone and other skeletal materials in 1963 and 1969.

Through the experience in analytical work on the Galley Hill, Swanscombe, Fontéchevade and Piltdown material (and wide-ranging comparative materials) it became an established procedure to analyse the following components of bone, antler, dentine and enamel: fluorine (F), phosphate (P_2O_5) and the ratio ($100F/P_2O_5$), calcium carbonate ($CaCO_3$), nitrogen (N) and uranium (either as U parts per million, as U_3O_8 ppm or as e U_3O_8 ppm). In the research carried out after 1960, the fluorine and phosphate contents were determined microchemically by methods developed by M. J. Glover and G. F. Phillips in the Laboratory of the Government Chemist, London. Where relative dating problems could be solved by estimating the fluorine content of the material by X-ray diffraction data in a long series of samples, the work was undertaken by G. F. Claringbull and R. J. Davis in the Department of Mineralogy at the Museum. Nitrogen determinations were made microchemically by E. I. Johnson in the Laboratory of the Government Chemist or by G. C. Ross in the Department of Zoology at the Museum, or by G. Weiler and F. B. Strauss in the Microanalytical Laboratory, Oxford. The uranium content of samples was determined in the Subdepartment of Anthropology at the Museum using the method of radiometric assay introduced by Davidson & Bowie (1955) during the Piltdown researches. Over 1200 radiometric assays were carried out in the Subdepartment of Anthropology between 1955 and 1975, the work being supervised first by A. E. Rixon and then mainly by Miss T. I. Molleson. When it was necessary to determine the uranium content on samples weighing less than a milligram, the work was generously undertaken by Dr R. L. Fleischer in the General Electric Research Laboratories at Schenectady, New York. (See Note on the Tables, p. 14).

In cases where it was desirable to have the chronometric age of a fossil hominid estimated as

closely as possible, the Research Laboratory of the British Museum (Bloomsbury) was asked to include one or more samples for radiocarbon dating in their 'human palaeontology' programme. I would like to acknowledge the help received from Messrs Harold Barker and Richard Burleigh of the British Museum Laboratory, in discussing problems of bone-dating relevant to the preparation of parts of the present paper.

The uses to which the research described in this paper can be put are quite varied. Perhaps partly as a result the use of fluorine, uranium and nitrogen in relative dating of bones which I have described in previous papers, a number of radiocarbon laboratories now include determinations of these elements in their reports. In the *Catalogue of Fossil Hominids* (BM(NH), London, 1967–1977), whenever they were obtainable, the results of analysing the hominid remains and of associated fauna have been recorded under item 9 as explained in the Introduction of each part of the Catalogue. The analyses were for the most part only obtainable through the co-operation of museum curators who had charge of the original specimens and who arranged for them to be lent for sampling at the BM(NH), or alternatively themselves supplied the requisite samples. Where analyses quoted in this Bulletin were made possible by co-operation of this kind, my indebtedness to the persons concerned has been expressed in the descriptions of the various sites.

Relative dating of skeletal material by nitrogen analysis has proved particularly useful when considered in relation to radiocarbon dating of specimens close to the Upper Palaeolithic/Holocene boundary (see under Robin Hood's Cave, p. 16). Since the breakdown of the collagen of skeletal materials into amino-acids, and the leaching out of these through weathering, have a bearing on the regression of the nitrogen content of buried bones, researches on these processes were carried out in the Department of Palaeontology in co-operation with other laboratories between 1953 and 1969. It is therefore appropriate to acknowledge here the help provided by the late Professor J. T. Randall and Miss A. V. W. Martin (now Mrs Angela Brown) in the Department of Biophysics, King's College London, who obtained the first electron micrographs of collagen fibrils in fossil ivory and fossil bone (p. 9). Further help on these lines was provided by D. Claugher, assisted by C. G. Ogden in the Electron Microscope Unit of the BM(NH), who in 1967 made a detailed study of the collagen fibres in the 30 000 years old rhinoceros bone from the Lloyd's site in the City of London (Fig. 5, p. 21). A related study was the identification of the amino-acids retained in the organic matrix of bones during their various stages of fossilization. This was undertaken by using chromatography, first by A. E. Rixon in the Department of Palaeontology using paper-chromatography, and later by G. C. Ross in the Department of Zoology, using a more sophisticated type of chromatography (Fig. 6, p. 21).

Further research is required on the ecological aspects of collagen degradation in buried skeletal materials. When ivory from ancient Egyptian sites is analysed prior to the use of any washing technique it is found to contain almost as much nitrogen as fresh ivory, and nearly the full range of the amino-acids. But if the analyses are carried out after a sample of the same ivory has been washed in warm (70°C) water and acetone, the nitrogen content has been greatly reduced and only a few of the amino-acids retained. Evidently under warm and relatively dry climatic conditions, the amino-acids in the collagenous residue are retained through many millennia, but they are loosely bound together, so that after washing in warm water they undergo dissolution. Buczko and his colleagues have found that the nitrogen content of bones buried in alluvial deposits in Hungary is lost most rapidly during warm climatic phases (Buczko *et al.* 1978). In Britain evidence has been found that the type of mineral matrix enclosing the fossil bones greatly affects the influence of the climatic factor in the rate of regression of nitrogen content. For example, the nitrogen content of the rhinoceros ulna encased by clay at the Lloyd's site in London was 3·4%, whereas in a mammoth bone embedded in sand on the same stratigraphic horizon at that site the nitrogen content had been reduced to 0·1%.

Altogether about a thousand skeletal samples ranging in age from Oligocene to late Holocene from sites in Africa, Asia, Australasia, Indonesia, Malaysia, North, Central and South America, and the Pacific Islands have been analysed. Dr Susan Limbrey, while working temporarily in the Subdepartment of Anthropology, BM(NH), prepared a slip index on which the essential details regarding each analysed sample are recorded, including the name of the analyst and the laboratory. Arrangements can be made for visitors to consult this data-bank when required.

From 1965 onwards Miss T. I. Molleson aided me in all phases of the programme of research described in this *Bulletin*. In thanking her for this co-operation, I should like to say how glad I am that since my retirement she has continued to use, and has made further developments in, the analytical studies of fossil skeletal materials which rather tentatively I initiated at the Museum some 30 years ago.

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